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### **Remedial Investigation Work Plan**

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

September 1, 2011

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### Remedial Investigation Work Plan

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Prepared for: National Grid

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### Acronyms and Abbreviations

ASP	Analytical Services Protocol
BTEX	benzene, toluene, ethylbenzene, and xylenes
BUG	Brooklyn Union Gas Company
CAMP	Community Air Monitoring Plan
CLP	Contract Laboratory Program
COCs	constituents of concern
COPCs	constituents of potential concern
CPP	Citizen Participation Plan
CSM	conceptual site model
CVOCs	chlorinated volatile organic compounds
DNAPL	dense non-aqueous phase liquid
DOT	Department of Transportation
DUSR	data usability summary report
EDD	Electronic Data Deliverable
EPA	United States Environmental Protection Agency
FSP	Field Sampling Plan
ft bls	feet below land surface
ft/d	feet per day
GPS	global positioning system

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HASP	Health and Safety Plan
HSA	hollow-stem auger
IDW	investigation-derived waste
KeySpan	KeySpan Corporation
LNAPL	light non-aqueous phase liquid
MGP	manufactured gas plant
msl	mean sea level
NAPL	non-aqueous phase liquid
National Grid	Brooklyn Union Gas d/b/a National Grid NY
NGVD	National Geodetic Vertical Datum
NTUs	nephelometric turbidity units
NYCRR	New York State Codes, Rules and Regulations
NYSDEC	New York State Department of Environmental Conservation
NYSDOH	New York State Department of Health
OD	outside diameter
PAHs	polycyclic aromatic hydrocarbons
PCBs	polychlorinated biphenyls
PCE	tetrachloroethene
PID	photoionization detector
PPE	personal protective equipment

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PVC	polyvinyl chloride
QA/QC	quality assurance/quality control
QAPP	Quality Assurance Project Plan
QHHEA	qualitative human health exposure assessment
RI	Remedial Investigation
SAP	Sampling and Analysis Plan
SC	Site Characterization
SCOs	soil cleanup objectives
SCGs	standards, criteria, and guidance
SOP	Standard Operating Procedure
SVOCs	semi-volatile organic compounds
TAL	Target analyte list
TCL	Target compound list
TOGS	Technical and Operational Guidance Series
TSDF	treatment, storage, and disposal facility
UST	underground storage tank
VAP	vertical aquifer profiling
VI	vapor intrusion
VOCs	volatile organic compounds

## Remedial Investigation Work Plan

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#### **Executive Summary**

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York. The Site occupies portions of two parcels located along Neptune Avenue and W. 5th Street. The Site location is shown on Figure 1.

The Site was operated by the Brooklyn Borough Gas Company, which was a predecessor company to National Grid. The Brooklyn Borough Gas Company was acquired by the Brooklyn Union Gas Company (BUG), which ultimately became KeySpan Corporation (KeySpan). KeySpan became National Grid following a merger in 2008.

A Site Characterization (SC) was conducted at the Site between November 2009 and March 2010. The findings of the SC are presented in the SC Data Summary and SC Data Summary Addendum. Based on the findings of the SC and a July 13, 2010 meeting between the New York State Department of Environmental Conservation (NYSDEC), National Grid, and ARCADIS, the RI will be conducted on two parcels (Block 7273, Lots 1 and 25) located along Neptune Avenue and W. 5th Street.

The investigation activities outlined in this RI Work Plan will provide data to address the following objectives:

- Determine the nature and extent of MGP-related constituents of concern (COCs) in soil, groundwater, and soil vapor on Lots 1 and 25, and if warranted based on the sampling data collected along the perimeters of Lots 1 and 25, expand the investigation beyond Lots 1 and 25. The majority of the RI will be conducted on Lots 1 and 25; two (2) water-table monitoring wells will be installed on Lot 50 to develop a further understanding of the groundwater flow regime.
- Determine the extent of MGP-related by-product residuals (e.g., coal tar, nonaqueous phase liquid [NAPL], purifier wastes, petroleum, solvents).
- Assess potential impacts to human health and the environment as a result of the release of COCs at the Site.

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The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5th Street to the east, a residential parcel to the south, and a commercial parcel to the west. Currently, the Site is developed with a shopping center and a parking lot for a high-rise apartment building.

Based on a review of available historical information, the Site was used as a MGP site from prior to 1895 until sometime between 1906 and 1930. The 1895 Sanborn map shows two gas holders, a retort house, two oil tanks, a tar tank, an engine room, a purifying house, and a shed. By 1906, the MGP Site was operated by the Brooklyn Borough Gas Company; an additional gas holder, generating house and cistern had been constructed, and the retort house and tar tank were no longer present. The MGP structures were removed sometime between 1906 and 1930. By 1930, the Site was occupied by a club house. By 1966, the Trump Village Shopping Center occupied the northern and central portions of the Site. Figure 2 shows the approximate location of the former MGP structures.

The data collected at the Site during the SC indicate that the former gas holders, tar tank, and cistern are all likely sources of the tar releases from the former MGP that were identified in the SC. Coal tar impacts were observed primarily in borings advanced in the central and eastern portions of the Site. The deepest tar-saturated soils were encountered in soil boring SB-1 in the 55 to 60 ft bls soil core.

Volatile organic compounds (VOCs) and semi-volatile organic compounds (SVOCs) were detected above standards, criteria, and guidance (SCGs) in soil and groundwater and were identified as COCs in these media. The highest benzene, toluene, ethylbenzene, and xylenes (BTEX) and polycyclic aromatic hydrocarbon (PAH) concentrations in soil generally correspond with the observed coal tar and petroleum impacts, which are a continuing source of groundwater impacts. The highest BTEX and PAH concentrations were detected in samples collected from monitoring well MW-5 (screened from 30 to 40 ft bls), indicating that dissolved-phase groundwater concentrations increase with depth within the zone of impacts.

The specific activities of the proposed RI are as follows:

 Drill and install four (4) water-table monitoring wells (MW-6 through MW-9) on Lots 25 and 50, as shown on Figure 3, to confirm the hydraulic gradient, and to develop a further understanding of the groundwater flow regime and how it influences contaminant transport.

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- Drill twelve (12) soil borings (SB-4 through SB-15) in the vicinity of the former MGP operations (Lots 1 and 25) to determine the three-dimensional extent of the NAPL impacts (see Figure 4). Eight (8) additional "step out" soil borings (SB-16 through SB-23) may be drilled if NAPL impacts are observed in the initial twelve (12) soil borings (see Figure 4). Soil samples will be collected and submitted to a laboratory for analysis as follows:
  - § Two (2) soil samples (1 soil sample per soil boring) will be collected from two (2) sonic soil borings (SB-4 and SB-5) and submitted to a laboratory for physical properties analyses (grain size analysis, pore fluid saturation, and water/NAPL relative permeability);
  - § Up to three (3) soil samples per soil boring will be collected and submitted to a laboratory for VOC, SVOC, target analyte list (TAL) metals, target compound list (TCL) polychlorinated biphenyls (PCBs), total cyanide, and free cyanide analyses. In addition, twenty percent of the soil samples collected from the soil borings will also be analyzed for TCL pesticides.

Three (3) groundwater samples will be collected from a temporary well in one (1) sonic boring (SB-7) and will be submitted to a laboratory for VOC and SVOC analyses. Three (3) monitoring wells (MW-10 through MW-12) will be installed in sonic soil borings SB-4, SB-5, and SB-7 and will be screened based on the observed NAPL distribution.

- Conduct vertical aquifer profiling (VAP) sampling in five (5) borings (VP-1 through VP-5) and collect fifteen (15) groundwater samples (3 groundwater samples per boring) for VOC and SVOC analyses (see Figure 4). The data will be used to determine the nature and distribution of the dissolved-phase groundwater impacts on Lots 1 and 25.
- Drill and install four (4) monitoring wells (MW-13 through MW-16) on Lots 1 and 25. The monitoring well locations and screen intervals will be determined based on the VAP groundwater quality data (VP-1 through VP-5).
- Drill four (4) temporary soil vapor points (SV-1 through SV-4 see Figure 5) on Lots 1 and 25 to collect soil vapor samples from the shallow vadose zone (5 ft bls).

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- Collect groundwater samples from the monitoring well network (MW-1 through MW-16). The groundwater samples collected from monitoring wells MW-1 through MW-16 will be analyzed for VOCs, SVOCs, TAL metals, and total cyanide. In addition, groundwater samples collected from 3 monitoring wells will also be analyzed for TCL PCBs and TCL pesticides (i.e., 20 percent of the 16 monitoring wells).
- Based on an evaluation of data collected above, assess the need for additional investigation efforts and, if necessary, identify additional investigation activities that may be needed to meet RI goals.

Upon completion of data collection and analysis, a qualitative human health exposure assessment (QHHEA) will be performed and an exposure assessment report will be prepared and included with the RI Report. The QHHEA will be conducted in accordance with NYSDEC DER-10 (NYSDEC, 2010). The purpose of the QHHEA is to determine whether Site conditions pose an unacceptable hazard to potentially exposed receptor populations. To pose an unacceptable hazard to receptor populations, the receptor must be exposed to contaminants at the Site. The QHHEA will evaluate whether complete exposure pathways exist at the Site and identify constituents of potential concern (COPCs) for those receptors and media of concern where a complete exposure pathway exists (NYSDEC, 2010).

Once sufficient information is collected to complete the RI and address the RI objectives, the RI Report will be prepared and submitted to the NYSDEC for review. The RI Report will incorporate the SC data generated prior to the RI, will be prepared following applicable NYSDEC guidance, and will be consistent with the requirements of the Order on Consent for the Site.

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### 1. Introduction

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York. The Site occupies portions of two parcels located along Neptune Avenue and W. 5th Street. The Site location is shown on Figure 1.

The Site was operated by the Brooklyn Borough Gas Company, which was a predecessor company to National Grid. The Brooklyn Borough Gas Company was acquired by the Brooklyn Union Gas Company (BUG), which ultimately became KeySpan Corporation (KeySpan). KeySpan became National Grid following a merger in 2008. The MGP operated from prior to 1895 until sometime between 1906 and 1930. The MGP structures were dismantled sometime between 1906 and 1930, and the Site was subsequently sold to and redeveloped by third parties.

This RI Work Plan has been prepared in accordance with the requirements of a Multi-Site Order on Consent and Administrative Settlement (Index # A2-0552-0606) that was entered into by KeySpan and the New York State Department of Environmental Conservation (NYSDEC) in February 2007.

A Site Characterization (SC) was conducted at the Site between November 2009 and March 2010 in accordance with the NYSDEC-approved SC Work Plan (ARCADIS, 2009) and the SC Work Plan Addendum – Vapor Intrusion Investigation (ARCADIS, 2010). The findings of the SC are presented in the SC Data Summary (ARCADIS, 2010a) and SC Data Summary Addendum (ARCADIS, 2010b). Based on the findings of the SC and a July 13, 2010 meeting between the NYSDEC, National Grid, and ARCADIS, the RI will be conducted on two parcels (Block 7273, Lots 1 and 25) located along Neptune Avenue and W. 5th Street. The RI will be conducted in a dynamic manner to allow flexibility in the technical approach via phasing of field efforts to maximize the effectiveness of data collection efforts.

### 1.1 Objectives

The investigation activities outlined in this RI Work Plan will provide data to address the following objectives:

• Determine the nature and extent of MGP-related constituents of concern (COCs) in soil, groundwater, and soil vapor on Lots 1 and 25, and if warranted

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based on the sampling data collected along the perimeters of Lots 1 and 25, expand the investigation beyond Lots 1 and 25. The majority of the RI will be conducted on Lots 1 and 25; two (2) water-table monitoring wells will be installed on Lot 50 to develop a further understanding of the groundwater flow regime.

- Determine the extent of MGP-related by-product residuals (e.g., coal tar, nonaqueous phase liquid [NAPL], purifier wastes, petroleum, solvents).
- Assess potential impacts to human health and the environment as a result of the release of COCs at the Site.

#### 1.2 Work Plan Organization

This RI Work Plan has been prepared in accordance with NYSDEC, Division of Environmental Remediation, *DER-10 Technical Guidance for Site Investigation and Remediation* (NYSDEC, 2010).

This RI Work Plan is organized into the following sections and appendices:

- Section 2 of this RI Work Plan summarizes the SC that was completed prior to the preparation of this RI Work Plan, the Site description and history, geology, and hydrogeology.
- Section 3 presents the preliminary conceptual site model (CSM).
- Section 4 discusses the applicable New York State standards, criteria, and guidance (SCGs).
- Section 5 discusses the approach and goals of the RI.
- Section 6 presents the proposed RI scope of work and the field activities to be conducted.
- Section 7 presents the Project Management Plan and anticipated schedule.
- Section 8 presents the references used to develop the RI Work Plan.

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- Appendix A DNAPL Contingency Plan describes detailed procedures to be followed during drilling to limit the potential for remobilization and downward migration of dense non-aqueous phase liquid (DNAPL).
- Appendix B Field Sampling Plan (FSP) describes detailed field procedures and protocols that will be followed during the field activities and provides standard operating procedures (SOPs).
- Appendix C Quality Assurance Project Plan (QAPP) presents the analytical methods and procedures that will be used to analyze soil, groundwater, soil vapor, and ambient (outdoor) air samples collected during the field activities.
- Appendix D Community Air Monitoring Plan (CAMP) presents air monitoring and response efforts to detect and mitigate potential airborne releases of COCs during the field activities.
- Appendix E Health and Safety Plan (HASP) presents the site-specific procedures to protect Site workers conducting the RI field activities.
- Appendix F Citizen Participation Plan (CPP) promotes communication among all parties involved with, or affected by, contamination at the Site.

### 2. Site Description and History

This section of the RI Work Plan presents the Site setting, a summary of the Site history, current Site conditions, and geologic and hydrogeologic conditions in the Site vicinity, and summarizes the SC that was completed prior to the preparation of this RI Work Plan. A detailed description of the Site history is provided in Section 2 of the SC Work Plan.

#### 2.1 Site Setting

The Site is located at 486 Neptune Avenue in the Borough of Brooklyn, New York City, Kings County, New York and occupies portions of two parcels that are identified by Tax Map Number: Block 7273, Lots 1 and 25. As shown on Figure 1, the Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is generally flat with an elevation of approximately 9 feet above mean sea level (msl). The closest natural surface water

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body is Coney Island Creek, which is located approximately 0.25 miles to the northwest of the Site.

The layout of the Site and surrounding properties is presented on Figure 2 (Site Plan). The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5th Street to the east, a residential parcel to the south, and a commercial parcel to the west. Currently, the Site is developed with a shopping center and a parking lot for a high-rise apartment building. Land use and zoning at the Site and the other properties in the area is commercial and residential. Land use to the north is residential and commercial, land use to the east and south is residential, and land use to the west is commercial (New York City Planning Commission Zoning Map, 2009).

#### 2.2 Site History

This section discusses the historical use of the Site, with emphasis on the former MGP operations. The information reviewed to produce this summary included:

- Sanborn fire insurance maps
- Aerial photographs (EDR, 2008)

An overview of the historical MGP operations is discussed below. A detailed timeline of key observations based on the review of historical information in connection with the Site and an overview of land use in the Site vicinity is presented in Section 2.2.2 of the SC Work Plan (ARCADIS, 2009).

Based on a review of available historical information, the Site was used as a MGP site from prior to 1895 until sometime between 1906 and 1930. The 1895 Sanborn map shows two gas holders, a retort house, two oil tanks, a tar tank, an engine room, a purifying house, and a shed. By 1906, the MGP Site was operated by the Brooklyn Borough Gas Company; an additional gas holder, generating house and cistern had been constructed, and the retort house and tar tank were no longer present. The MGP structures were removed sometime between 1906 and 1930. By 1930, the Site was occupied by a club house. By 1966, the Trump Village Shopping Center occupied the northern and central portions of the Site. Figure 2 shows the approximate location of the former MGP structures.

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### 2.3 Current Conditions

As discussed above, the Site is currently occupied by a shopping center and a parking lot for a high-rise apartment building. The eastern portion of the shopping center is situated above the former MGP structures. As shown on Figure 2, the majority of the Site is either paved or developed with buildings.

Current shopping center occupants that are situated in the vicinity of/above the approximate location of the former MGP structures (i.e., eastern portion of shopping center) are as follows:

- Radio Shack
- Silent Thunder Martial Arts
- West 5<sup>th</sup> Medical Supply
- Eastern Chinese Restaurant
- Kurt Cleaners
- Capital One Bank
- CVS Pharmacy

Based on a regulatory database search conducted by GEI Consultants, Inc. (GEI, 2007), environmental records information for the Site indicated the following:

- Two (2) No. 2 fuel oil underground storage tanks (USTs) were discovered to be leaking based on tank testing. The USTs are located at 2928 W. 5<sup>th</sup> Street, which is the address of the residential parcel (Block 7273, Lot 25) on which the southern portion of the Site is located.
- Kurt Cleaners (current shopping center occupant) was identified as a small quantity generator.

No environmental records information was identified for properties abutting Lots 1 and 25 of Block 7273.

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#### 2.4 Geology

This section of the RI Work Plan describes the regional and local geology.

#### 2.4.1 Regional Geology

The unconsolidated geologic deposits underlying Kings County consist of clay, silt, sand, and gravel that overlie southward-dipping consolidated bedrock. The crystalline bedrock consists mainly of Precambrian age granite, gneiss, and schist. The overlying unconsolidated sediments were deposited during the Cretaceous and form, in ascending order, the Raritan and Magothy Formations. During the Pleistocene, several episodes of glaciation eroded the Cretaceous deposits (Smolensky, et al, 1989). The oldest Pleistocene deposit is the Jameco Gravel (Jameco aquifer), which overlies the Magothy Formation and Raritan confining unit and is present only in western Long Island. The Gardiners Clay overlies the Jameco Gravel, Magothy Formation, and Raritan confining unit in western Long Island. The Upper Pleistocene deposits formed when the glacial ice and glacial meltwater deposited till and outwash material, forming what is presently known as the Upper Glacial aquifer.

The Raritan Formation consists of the Lloyd Sand and the Raritan Clay. The Lloyd aquifer (the hydrogeologic equivalent of the Lloyd Sand) consists of fine to coarse sand, gravel, commonly with a clayey matrix, and lenses and layers of silty and solid clay. The Raritan confining unit (the hydrogeologic equivalent of the Raritan Clay) is regionally continuous and consists of silty and solid clay, and lenses and layers of sand. Because of its low permeability, the Raritan Clay serves as a confining unit for the underlying Lloyd Sand.

The Magothy Formation is a deltaic deposit consisting of fine to medium sand, clayey in part, interbedded with lenses and layers of coarse sand, silt, and sandy and solid clay. Gravel is common in the basal zone of the Magothy Formation.

The Jameco aquifer (the hydrogeologic equivalent of the Jameco Gravel) is a channel filling consisting of fine to very coarse sand and gravel with few layers of clay and silt (Smolensky, et al, 1989).

The Gardiners Clay is a lagoonal/shallow-bay clay consisting of clay, silt, and few layers of sand and gravel (Smolensky, et al, 1989).

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The Upper Glacial aquifer consists primarily of till and glacial outwash deposits. The till, composed of clay, sand, gravel, and boulders, forms the Harbor Hill and Ronkonkoma terminal moraines. These terminal moraines represent the farthest advance of late-Pleistocene glaciation on Long Island. South of the morainal deposits is a glacial outwash plain, which, in Kings County, extends from the Harbor Hill moraine to Jamaica Bay and New York Bay, and consists of fine to very coarse sand and pebble to boulder sized gravel (Smolensky, et al, 1989).

#### 2.4.2 Local Geology

The Site is located south of the Harbor Hill terminal moraine and the surficial deposits consist of glacial outwash deposits (Upper Glacial aquifer) at the Site. Based on a review of the U.S. Geological Survey publication entitled *Hydrologic Framework of Long Island, New York, U.S. Geological Survey Hydrologic Investigations Atlas HA-709* (Smolensky, et al, 1989), bedrock beneath the Site is found at an approximate elevation of 650 feet below msl. The Lloyd aquifer, which overlies bedrock, has a surface elevation of approximately 500 feet below msl. The Raritan Clay has a surface elevation of approximately 250 feet below msl. The Jameco aquifer has a surface elevation of approximately 200 feet below msl. The Gardiners Clay has a surface elevation of approximately 150 feet below msl. The Upper Glacial aquifer corresponds to the saturated upper part of the highly permeable Pleistocene deposits of sand and gravel.

Based on the soil borings that were drilled during the SC, primarily fine to coarse sand deposits (glacial outwash deposits [Upper Glacial aquifer]) were encountered during the subsurface investigation. Approximately 5 to 7 feet of fill material was encountered in the upper portion of the soil borings. No confining layers were observed during the SC drilling activities.

### 2.5 Hydrogeology

The principal aquifers underlying the project area are the Upper Glacial aquifer, Jameco aquifer, and Magothy aquifer. The Gardiners Clay hydraulically confines the Magothy and Jameco aquifers in most of Kings County; the Jameco aquifer and Magothy aquifer hydrogeologic units are in direct hydraulic connection with each other. Groundwater in the Upper Glacial aquifer occurs under unconfined conditions at and near the Site. Within the project area, the average horizontal hydraulic conductivity of the Upper Glacial aquifer is approximately 270 feet per day (ft/d), with an anisotropy

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ratio of approximately 10:1 (horizontal to vertical, respectively) (McClymonds and Franke, 1972). The average horizontal hydraulic conductivity of the Jameco aquifer in the project area is approximately 200 to 300 ft/d, with an anisotropy ratio of approximately 10:1 (horizontal to vertical, respectively) (McClymonds and Franke, 1972). The average horizontal hydraulic conductivity of the Magothy aquifer in the project area is approximately 50 ft/d, with an anisotropy ratio of approximately 100:1 (horizontal to vertical, respectively) (McClymonds and Franke, 1972).

The Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. Based on the SC, the shallow groundwater flow direction is generally to the northwest, toward Coney Island Creek. The Site is also located north of a major groundwater discharge boundary (i.e., New York Bay) and a local groundwater divide may be influencing groundwater flow and contaminant transport. The depth to groundwater at the Site is approximately 8 feet below land surface (ft bls).

#### 2.6 Previous Investigations

This section of the RI Work Plan summarizes the previously completed SC investigation at the Site. The SC data have been considered during the preparation of this RI Work Plan and will be documented in the final RI Report (see Section 6.2.7 of this RI Work Plan). The SC investigation activities and findings are presented in the SC Data Summary (ARCADIS, 2010a) and SC Data Summary Addendum (ARCADIS, 2010b).

The SC investigation involved the following activities:

- A soil investigation including the drilling of soil borings and the collection of subsurface soil samples for analysis.
- A groundwater investigation including collection of a groundwater sample from a temporary monitoring well, installation of groundwater monitoring wells, characterization of groundwater flow and quality, and determination of the presence/absence of NAPL.
- A vapor intrusion (VI) investigation including the installation of temporary subslab soil vapor points and characterization of sub-slab soil vapor quality, ambient air quality sampling, and indoor air quality sampling.

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The key findings of the SC were as follows:

- The primary volatile organic compounds (VOCs) that were detected in soil above SCGs include benzene, toluene, ethylbenzene, and xylenes (BTEX). VOCs were identified as COCs in soil.
- The primary semi-volatile organic compounds (SVOCs) that were detected in soil above SCGs include polycyclic aromatic hydrocarbons (PAHs). SVOCs were identified as COCs in soil.
- Pesticides are not associated with former MGP operations and were not identified as COCs in soil relative to the former MGP site investigation. Nonetheless, a subset (i.e., 20 percent) of the soil samples collected during the RI will be analyzed for pesticides.
- Polychlorinated biphenyls (PCBs) were not identified as COCs in soil. Nonetheless, the soil samples collected during the RI will be analyzed for PCBs.
- Three (3) metals (manganese, selenium, and mercury) were detected in soil above SCGs. These metals may be associated with former MGP operations, but may be present at the Site due to the post-MGP placement of fill material. Metals were identified as potential MGP-related COCs in soil.
- Total cyanide was detected in soil above its SCG in one soil sample; as noted above, cyanide may be associated with the post-MGP placement of impacted fill material. Free cyanide was only detected in one soil sample. Total cyanide was identified as a potential MGP-related COC in soil.
- The primary VOCs and SVOCs that were detected in groundwater above SCGs include BTEX and PAHs, respectively. VOCs and SVOCs were identified as COCs in groundwater.
- Pesticides, metals, and cyanide were not identified as COCs in groundwater. Nonetheless, the groundwater samples collected during the RI will be analyzed for metals and cyanide; a subset (i.e., 20 percent) of the groundwater samples will be analyzed for pesticides.

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- PCBs were not detected in groundwater above the laboratory reporting limits. Nonetheless, a subset (i.e., 20 percent) of the groundwater samples collected during the RI will be analyzed for PCBs.
- Neither light non-aqueous phase liquid (LNAPL) nor DNAPL were detected in any of the monitoring wells.
- The hydrocarbon product identification data for soil samples suggest that fuel oil impacts of unknown origin are present at the water table across the entire area that the former MGP occupied and that coal tar impacts are present at the water table across the central and eastern portion of the area that the former MGP occupied.
- The former gas holders, tar tank, and cistern are all likely sources of the tar releases from the former MGP. Tar-saturated soils were observed in the glacial outwash deposits underlying the Site. The deepest tar-saturated soils were encountered in soil boring SB-1 in the 55 to 60 ft bls soil core.
- The highest BTEX and PAH concentrations in soil generally correspond with the observed tar and petroleum impacts, which are a continuing source of groundwater impacts.
- The extent to which elevated concentrations of BTEX and light-end PAH compounds in groundwater have migrated along the groundwater flow path (northwest) is unknown.
- Further investigation is required to characterize the distribution of MGP residuals on Lot 1 and the adjacent parcel to the south (Lot 25), and to delineate the lateral and vertical extent of groundwater impacts downgradient (i.e., northwest) of the former MGP.
- Potential MGP-related constituent vapors are not migrating into the shopping center building at concentrations that may result in an unacceptable human health risk. This is evidenced by the fact that potential MGP-related constituents detected in indoor air were below typical background indoor air concentrations for all indoor air quality samples. Furthermore, the potential MGP-related constituents detected in indoor air may be attributable to other sources (i.e., background sources).

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Tetrachloroethene (PCE) and other chlorinated VOCs (trichloroethene [TCE], cis-1,2-dichloroethene [cis-1,2-DCE], and vinyl chloride [VC]) were detected at elevated concentrations in the SSSV-6 sub-slab soil vapor sample. A number of non-MGP-related constituents detected in indoor air (2-butanone [methyl ethyl ketone], dichlorodifluoromethane (Freon 12), 4-methyl-2-pentanone [MIBK], and PCE) were above typical background indoor air concentrations in a number of indoor air quality samples.

### 3. Preliminary Conceptual Site Model

This section of the RI Work Plan discusses the preliminary CSM for the Site. The purpose of the preliminary CSM is to describe the release(s) of COCs to the environment, and the nature and degree of the release. The preliminary CSM also serves as the basis for determining additional site characterization needs; therefore, the preliminary CSM is the basis for the scope of work that is presented in this RI Work Plan.

Fill material placed on the Site after the dismantling of structures and sale of the parcel (as evidenced by the presence of pesticides not yet developed circa 1930) was encountered in the upper 5 to 7 feet in soil borings. Based on the presence of pesticides in the fill material, it is believed that the metals impacts in shallow soils are also associated with impacted historic fill. The metals that were detected in the fill material at concentrations that slightly exceeded the protection of groundwater soil cleanup objectives (SCOs) include selenium (SB-1 [2-3']) and mercury (SB-2 [2-3']). However, the concentrations of selenium and mercury were below the protection of public health commercial use SCO and were not detected in groundwater. Therefore, metals are not considered COCs in soil. Total cyanide exceeded the protection of public health commercial use SCO in one soil sample (SB-1 [2-3']). Based on the total cyanide concentration (35,200 micrograms per kilogram [µg/kg]) detected in the SB-1 (2-3') soil sample, cyanide is considered to be a COC in soil and will be further investigated during the RI.

The data collected at the Site during the SC indicate that the former gas holders, tar tank, and cistern are all likely sources of the tar releases from the former MGP that were identified in the SC. Coal tar impacts were observed primarily in borings advanced in the central and eastern portions of the Site. The deepest tar-saturated soils were encountered in soil boring SB-1 in the 55 to 60 ft bls soil core. It is likely that coal tar encountered the water table in the vicinity of the former gas holders and tar handling structures, spread out laterally after encountering the water table, and then

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penetrated into the unconfined saturated zone. The tar released from the MGP sources generally continued to migrate downward through the saturated permeable glacial outwash deposits until the volume of tar was insufficient to maintain a DNAPL fluid pressure capable of overcoming the pressure of the surrounding water (i.e., the DNAPL pressure head no longer exceeded the capillary pressure, which impeded further downward migration of the tar).

Hydrocarbon identification analyses were conducted for soil samples collected from the water table interface (7 to 9 ft bls) to further support the evaluation of subsurface impacts. The chromatograms for samples collected from the western portion of the Site (i.e., MW-1, MW-3, and SB-1 locations) have a carbon range similar to that of #4 and #6 fuel oils; the chromatograms for samples collected from the eastern/central portions of the Site (i.e., MW-2, MW-4, and SB-2 locations) have a carbon range similar to that of #4 and #6 fuel oils and also showed similarities to coal tar. These data suggest that fuel oil impacts of unknown origin are present at the water table across the entire area that the former MGP occupied, and further support the premise that coal tar (DNAPL) was primarily released beneath the central and eastern portion of the area that the former MGP occupied.

VOCs and SVOCs were detected above SCGs in soil and groundwater and were identified as COCs in these media. The highest BTEX and PAH concentrations in soil generally correspond with the observed coal tar and petroleum impacts, which are a continuing source of groundwater impacts. As the groundwater flows in a northwesterly direction through these areas of coal tar impacts, dissolution of BTEX and light-end PAH compounds from the NAPL, along with their desorption from the aquifer matrix, will continue to persist and act as a source of groundwater impacts. The highest BTEX and PAH concentrations were detected in samples collected from monitoring well MW-5 (screened from 30 to 40 ft bls), indicating that dissolved-phase groundwater concentrations increase with depth within the zone of impacts. Monitoring well MW-5 was installed at the soil boring SB-3 location due to the observation of MGP-related impacts. The extent to which elevated concentrations of BTEX and light-end PAH compounds in groundwater have migrated along the groundwater flow path (northwest) is unknown.

This preliminary CSM will be re-evaluated and revised (as needed) as additional data are collected for the Site.

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#### 4. Standards, Criteria and Guidance

SCGs have been identified for the Site that pertain to meeting applicable regulations and RI objectives.

The SCGs for the Site soils are based upon the selection of applicable values from the New York State Codes, Rules and Regulations Title 6 (6 NYCRR) Part 375 Remedial Program Restricted Use SCOs. The applicable SCGs are as follows:

- Protection of public health commercial use SCOs; and,
- Protection of groundwater SCOs

The SCGs for groundwater are based on the NYSDEC Division of Water Technical and Operational Guidance Series (TOGS) (1.1.1) Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations.

No SCGs currently exist for exterior soil vapor.

### 5. RI Work Plan Approach and Goals

This section of the RI Work Plan describes the approach, rationale, and goals for the RI.

#### 5.1 Remedial Investigation Approach

To successfully meet the RI objectives in an effective manner, additional data will be collected during the RI. The data collected during the SC investigation provided an initial characterization of the existing conditions.

The existing SC data were used to develop the RI scope of work proposed in this RI Work Plan. This approach ensures that the most complete and recent data set is embedded within the decision-making process so that a complete and focused RI is performed and provides a sound technical basis for the field efforts based on the best available data.

In particular, this RI Work Plan provides a detailed scope of work for investigating soil, groundwater, and soil vapor on Lots 1 and 25 to meet the stated goals of the RI. In addition, two (2) water-table monitoring wells will be installed on Lot 50. The RI data

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will be evaluated after it has been received, reviewed, and validated. However, prior to the data validation process, unvalidated data will be provided to the NYSDEC, as necessary, to allow for a timely review and determination as to whether additional investigative activities are required. Should the RI data indicate further investigative activities (e.g., groundwater beyond Lots 1, 25, and 50) are necessary to meet the RI goals, a supplemental RI Work Plan, in the form of a focused letter detailing proposed additional investigative activities and rationale, will be submitted to the NYSDEC. Additional work, if necessary, will be implemented with NYSDEC approval.

The decision-making process for the RI activities is as follows:

- Install water-table monitoring wells MW-6 through MW-9 on Lots 25 and 50 to confirm the hydraulic gradient (observed during the SC to be very flat), and to develop a further understanding of the groundwater flow regime and how it influences contaminant transport. The water-level data collected from these monitoring wells will aid in determining if additional investigative activities (e.g., groundwater sampling beyond Lots 1, 25, and 50) are warranted.
- Drill soil borings SB-4 through SB-15. If warranted, based on the inspection of soil cores from soil borings SB-4 through SB-15 (see Section 6.2.1.1 of this RI Work Plan), additional soil borings SB-16 through SB-23 ("step out" soil borings) will be drilled to delineate the NAPL impacts.
- Drill temporary monitoring wells (vertical aquifer profiling [VAP] borings) VP-1 through VP-5 for the collection of groundwater quality data.
- Additional monitoring wells (MW-13 through MW-16) will be installed to monitor groundwater quality over time based on the temporary monitoring wells (VAP borings) groundwater quality data. The locations of these additional monitoring wells will be determined after the VAP boring data have been evaluated and will be discussed with and approved by the NYSDEC.
- Soil vapor sampling (SV-1 through SV-4) will be conducted in accordance with the New York State Department of Health (NYSDOH) Guidance for Evaluating Soil Vapor Intrusion in the State of New York (NYSDOH, 2006).
- If warranted, based on the groundwater quality data, additional investigative activities (e.g., groundwater sampling beyond Lots 1, 25, and 50) will be

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identified and performed upon receiving NYSDEC approval, as described above.

### 5.2 RI Goals

The following are the specific goals of the RI:

- Characterize soil quality and determine the nature and extent of MGP-related COCs.
- Characterize groundwater quality and determine the nature and extent of MGP-related COCs.
- Characterize shallow (i.e., approximately 5 ft bls) soil vapor quality.
- Fully develop the list of COCs for the Site.
- Characterize the risks posed by affected media.
- Determine potential receptors and exposure pathways associated with potential exposure to soil, groundwater, and soil vapor.
- Determine if additional data collection efforts are warranted to meet RI goals based on the data collected on Lots 1, 25, and 50. If it is determined that additional data collection efforts are warranted beyond the work described in Section 6.2.1 of this RI Work Plan, then additional investigation activities that may be needed to meet RI goals will be identified, as described in Section 5.1 of this RI Work Plan.
- Obtain sufficient information to evaluate the necessity for further action.

### 6. RI Tasks

This section describes the proposed RI scope of work.

#### 6.1 Scoping the RI

The scoping process, for the purpose of identifying and defining the RI tasks described below, included the following:

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- Visits to the Site.
- Evaluation of the Order on Consent requirements and relevant State and Federal guidance documents.
- Evaluation of existing reports and data (i.e., SC Data Summary and SC Data Summary Addendum) for the Site.

#### 6.2 Remedial Investigation

This section of the RI Work Plan describes the proposed RI scope of work. The proposed RI scope of work includes investigation on Lots 1, 25, and 50, and, if warranted based on the investigation data, investigation beyond Lots 1, 25, and 50 (see Section 5.1 of this Work Plan). Table 1 and Figures 3 through 5 of this RI Work Plan provide additional information and the rationale for the scope of work presented in this section.

Field sampling, laboratory analysis, and field work for the RI will be conducted in accordance with the protocols described in the Sampling and Analysis Plan (SAP), as described below and documented, in detail, in Appendix A (DNAPL Contingency Plan), Appendix B (FSP), and Appendix C (QAPP). Additional plans that will be followed during the RI are provided in Appendix D (CAMP), Appendix E (HASP), and Appendix F (CPP).

The specific activities of the proposed RI are as follows:

- Drill and install four (4) water-table monitoring wells (MW-6 through MW-9) on Lots 25 and 50, as shown on Figure 3, to confirm the hydraulic gradient (observed during the SC to be very flat), and to develop a further understanding of the groundwater flow regime and how it influences contaminant transport. The wells will be installed using hollow-stem auger (HSA) drilling techniques.
- Drill twelve (12) soil borings (SB-4 through SB-15) in the vicinity of the former MGP operations (Lots 1 and 25) to determine the three-dimensional extent of the NAPL impacts (see Figure 4). The borings will be advanced using a combination of direct push (8 borings) and sonic (4 borings) drilling techniques. Eight (8) additional "step out" soil borings (SB-16 through SB-23) may be drilled if NAPL impacts are observed in the initial twelve (12) soil borings (see

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Figure 4). Soil samples will be collected and submitted to a laboratory for analysis as follows:

- § Two (2) soil samples (1 soil sample per soil boring) will be collected from two (2) sonic soil borings (SB-4 and SB-5) and submitted to a laboratory for physical properties analyses (grain size analysis, pore fluid saturation, and water/NAPL relative permeability);
- § Up to three (3) soil samples per soil boring will be collected and submitted to a laboratory for VOC, SVOC, target analyte list (TAL) metals, target compound list (TCL) PCBs, total cyanide, and free cyanide analyses. In addition, twenty percent of the soil samples collected from the soil borings will also be analyzed for TCL pesticides.

Three (3) groundwater samples will be collected from a temporary well in one (1) sonic boring (SB-7) and will be submitted to a laboratory for VOC and SVOC analyses. Three (3) monitoring wells (MW-10 through MW-12) will be installed in sonic soil borings SB-4, SB-5, and SB-7 and will be screened based on the observed NAPL distribution. Monitoring wells MW-10 through MW-12 will be installed with 3-foot long sumps.

- Conduct VAP sampling in five (5) borings (VP-1 through VP-5) using direct push drilling techniques and collect fifteen (15) groundwater samples (3 groundwater samples per boring) for VOC and SVOC analyses (see Figure 4). The data will be used to determine the nature and distribution of the dissolvedphase groundwater impacts on Lots 1 and 25.
- Drill and install four (4) monitoring wells (MW-13 through MW-16) on Lots 1 and 25 using sonic drilling techniques. The monitoring well locations and screen intervals will be determined based on the VAP groundwater quality data (VP-1 through VP-5).
- Drill four (4) temporary soil vapor points (SV-1 through SV-4 see Figure 5) on Lots 1 and 25 to collect soil vapor samples from the shallow vadose zone (5 ft bls).
- Collect groundwater samples from the monitoring well network (MW-1 through MW-16). The groundwater samples collected from monitoring wells MW-1

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through MW-16 will be analyzed for VOCs, SVOCs, TAL metals, and total cyanide. In addition, groundwater samples collected from 3 monitoring wells will also be analyzed for TCL PCBs and TCL pesticides (i.e., 20 percent of the 16 monitoring wells).

 Based on an evaluation of data collected above, assess the need for additional investigation efforts and, if necessary, identify additional investigation activities that may be needed to meet RI goals, as described in Section 5.1 of this RI Work Plan.

#### 6.2.1 Proposed Remedial Investigation

The following subsections of this RI Work Plan describe, in detail, the rationale for the proposed RI scope of work. The scope of work is presented in detail in Table 1 and on Figures 3 through 5. Detailed procedures to limit the potential for remobilization and downward migration of DNAPL are provided in Appendix A of this RI Work Plan (DNAPL Contingency Plan). Detailed field methodologies and SOPs are provided in Appendix B of this RI Work Plan (FSP). Quality assurance/quality control (QA/QC) procedures and protocols, analyte lists, analytical methods, and sample handling procedures are provided in Appendix C of this RI Work Plan (QAPP). Air monitoring and response efforts to protect the downwind community during the field activities are provided in Appendix D of this RI Work Plan (CAMP). Health and safety procedures are provided in Appendix E of this RI Work Plan (HASP). Community outreach and participation activities are provided in Appendix F of this RI Work Plan (CPP).

Field personnel will mobilize to the Site to verify existing Site conditions and label and/or mark the proposed sample or monitoring well locations shown on Figures 3 through 5. Once the sample locations are marked, New York's DigNet of New York City & Long Island will be contacted to mark underground utilities in areas where intrusive activities (i.e., drilling, soil sampling, well installation, soil vapor sampling) will occur. The Site property owners, adjacent property owners and/or private vendors will be contacted for assistance with mark out of utilities. Once the utilities are marked, equipment and personnel necessary to accomplish the RI activities will be mobilized to the Site. Given the numerous utilities likely present in an urban setting, the subsurface sample locations will be cleared of utilities to a depth of 5 ft bls by soft dig techniques (e.g., hand excavation, air knife).

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#### 6.2.1.1 Proposed Soil Investigation

Based on the results of the SC soil sampling program (see Section 2.6 of this RI Work Plan), the RI has been developed to meet the previously stated RI objectives. The proposed RI soil investigation to be performed at the Site includes subsurface soil sampling.

The proposed soil boring locations (SB-4 through SB-23) are shown on Figure 4. The actual drilling locations may be adjusted based on accessibility and field conditions (e.g., utilities), and in consultation with National Grid and the NYSDEC. A description of each soil boring to be drilled during the RI, as well as the corresponding soil samples and constituents to be analyzed, is provided in Table1. The detailed rationale for the RI soil borings is described below. The soil borings will be drilled using direct push (Geoprobe® or equivalent equipment) or sonic drilling techniques. Continuous soil sampling will commence at 5 ft bls. For each soil boring, the location will be determined relative to the New York State Plane Coordinate System using a global positioning system (GPS).

If a soil boring will be drilled using direct push drilling techniques, the Geoprobe® Dual Tube Sampling System will be used. The Geoprobe® Dual Tube Sampling System employs an outer casing and inner rod string assembly. Soil sampling will be conducted at the boring locations by advancing 3.25-inch outside diameter (OD) probe rods (outer casing) and a 5-foot long sample liner. Dual tube sampling uses two sets of probe rods to collect continuous soil cores. One set of rods is driven into the ground as an outer casing. These rods receive the driving force from the hammer and provide a sealed borehole from which soil samples may be collected. The second, smaller set of rods (inner rod string) are placed inside the outer casing. The inner rod string hold the sample liner in place as the outer casing is driven over the sampling interval. The inner rod string is then retracted to retrieve the filled sample liner.

If using sonic drilling techniques, soil sampling will be conducted at the boring locations by driving a 5-foot long 4-inch diameter core barrel using vibration, rotation, and a downward force. Once the core barrel has been advanced, a 6-inch diameter secondary or "override" casing will be advanced down to the same depth as the inner core barrel. The override casing keeps the borehole from collapsing while the inner core barrel is removed. Once the core barrel is removed, the soil core will be extruded from the core barrel.

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Soil recovered from each sample interval will be visually characterized for color, texture, and moisture content as described in the National Grid *Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites* (Appendix B [FSP] of this RI Work Plan). The presence of visible staining, NAPL, and obvious odors will be noted. If NAPL is encountered in any of the soil borings, the DNAPL Contingency Plan provided in Appendix A of this RI Work Plan will be implemented to limit the potential for remobilization and downward migration of DNAPL.

The soil borings will be drilled to a target depth of 80 ft bls. If evidence of MGP-related impacts is observed, the boring will continue to approximately 10 feet beyond the observed impacts for vertical delineation purposes, until refusal is encountered, or until a confining layer is observed. It is anticipated that a depth of 80 ft bls is an appropriate target depth to delineate the vertical extent of NAPL impacts based on the SC data. At three of the four sonic soil boring locations (SB-4, SB-5, and SB-7), a monitoring well will be installed, as described in Section 6.2.1.2.2 of this RI Work Plan. The SB-4, SB-5, and SB-7 soil borings will be sealed (with a cement/bentonite grout or bentonite pellets) from the terminal depth of the boring up to the planned depth of the monitoring well and the monitoring wells will be installed. Soil borings SB-6 and SB-8 through SB-23 will be grouted from the terminal depth of the boring to land surface.

Air monitoring will be conducted in the worker breathing zone during implementation of the RI work activities (most notably during the drilling activities). The air will be monitored using a photoionization detector (PID), a multi-gas meter, and a real-time aerosol monitor. Additional perimeter air monitoring to be conducted at the boundary of the work area during the investigation is detailed in the CAMP (Appendix D of this RI Work Plan). Appendix E of this RI Work Plan (HASP) provides the health and safety procedures that will be implemented to protect Site workers conducting the RI field activities.

#### 6.2.1.1.1 Subsurface Soil Analyses

Up to three (3) subsurface soil samples will be collected from soil borings SB-6 through SB-23 and will be submitted to the laboratory for the analysis of VOCs, SVOCs, TAL metals, TCL PCBs, total cyanide, and free cyanide (analysis by EPA Method 9016) (see QAPP for compound lists). Twenty percent of the soil samples collected from the soil borings will also be analyzed for TCL pesticides. One sample will be collected from the depth interval where the greatest apparent degree of impacts is observed, and a third soil

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sample ("un-impacted" soil) may also be collected below apparent "impacted" soil to aid in vertical delineation, if warranted. The field representative will select soil samples to meet the RI objectives based on visual observation of staining in the soil sample interval and/or the sample interval with the highest PID reading. If no staining or PID readings are encountered at a boring location, the second soil sample will be collected immediately above the groundwater table for laboratory analysis. At locations where the intent of the soil boring is to vertically delineate NAPL impacts (SB-4 and SB-5) that were identified during the SC, one (1) soil sample ("un-impacted" soil) will be collected at a depth 10 feet below apparent "impacted" soil to aid in vertical delineation.

Soil samples will be collected following the procedures outlined in the ARCADIS *Soil Drilling and Sample Collection* SOP (provided in Appendix B [FSP] of this RI Work Plan) and as described in this Section of the RI Work Plan. The soil cores will be screened for organic vapors using the jar headspace method and a PID following the procedures outlined in the ARCADIS Photoionization Detector Air Monitoring and Field *Screening* SOP (provided in Appendix B [FSP] of this RI Work Plan). Soil samples will be collected from the appropriate two-foot interval using a decontaminated stainless steel spoon or trowel. The VOC sample will be immediately transferred directly into the laboratory-supplied sample bottles. A sufficient amount of the remaining soil will be homogenized by mixing the sample in a decontaminated stainless steel bowl with a decontaminated stainless steel spoon or trowel following the procedures outlined in the ARCADIS *Compositing or Homogenizing Samples* SOP (provided in Appendix B [FSP] of this RI Work Plan). Laboratory-supplied sample containers for other analytes (i.e., SVOCs, TAL metals, TCL PCBs, total cyanide, free cyanide, and TCL pesticides) will then be filled. All sample bottle caps will be secured snugly, but not over-tightened.

Samples will be submitted to a NYSDOH accredited laboratory certified for the selected analysis. Analytical methods, sample handling, and laboratory protocols are outlined in the QAPP (Appendix C of this RI Work Plan). Sample analyses will follow the NYSDEC Analytical Services Protocol (ASP) (most recent version) and will include QA/QC samples at a frequency indicated in the QAPP. Analytical results for analysis of the soil samples will be reported using NYSDEC ASP Category B data deliverables.

Equipment decontamination will follow the procedures outlined in the ARCADIS *Field Equipment Decontamination* SOP (provided in Appendix B [FSP] of this RI Work Plan). In general, non-disposable equipment, including drilling tools and equipment, will be decontaminated prior to first use on Site, between each investigation location, and prior to demobilization (if dedicated equipment is not used). The integrity of the

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decontamination procedures will be checked periodically with equipment rinse blanks, as required by the QAPP.

Investigation-derived waste (IDW) will be containerized in appropriate waste containers and staged on site prior to off-site disposal following the procedures outlined in the ARCADIS *Investigation-Derived Waste Handling and Storage* SOP (provided in Appendix B [FSP] of this RI Work Plan). Soil cuttings, personal protective equipment (PPE), and spent disposable sampling materials will be segregated by waste type and placed in Department of Transportation (DOT)-approved 55-gallon steel drums. Decontamination water and drilling water will be stored in DOT-approved 55-gallon steel drums or a polyethylene storage tank. Storage vessels will be appropriately labeled with the contents, generator, location, and date for later off-site transportation and disposal by National Grid.

#### 6.2.1.2 Proposed Groundwater Investigation

The proposed groundwater RI consists of the advancement of five (5) temporary monitoring wells (VAP borings VP-1 through VP-5), pneumatic slug testing, the installation of eleven (11) additional monitoring wells on Lots 1, 25, and 50 (MW-6 through MW-16), and the sampling of the sixteen (16) monitoring wells. The existing data collected and interpretations made to this point were used to determine the best locations for the temporary monitoring wells proposed herein. The locations of the proposed temporary monitoring wells and permanent monitoring wells MW-6 through MW-12 are shown on Figures 3 and 4. It is anticipated that monitoring wells MW-13 through MW-15 will be installed in the vicinity of temporary monitoring wells VP-1 and VP-3 and that monitoring well MW-16 will be installed adjacent to water-table monitoring well MW-7. Table 1 provides the complete description/rationale for the proposed scope of work.

#### 6.2.1.2.1 Temporary Monitoring Wells

Groundwater samples will be collected from temporary monitoring wells VP-1 through VP-5 (direct push VAP borings) and a temporary monitoring well that will advanced at the SB-7 location (sonic boring). The groundwater samples will be submitted to the laboratory for the analysis of VOCs and SVOCs. The intent of these groundwater samples is to determine the nature and distribution of the dissolved-phase groundwater impacts on Lots 1 and 25. For each temporary monitoring well, the location will be determined relative to the New York State Plane Coordinate System using a GPS.

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It is anticipated that the temporary monitoring well groundwater samples will be collected from three (3) depth intervals as follows:

- 13 to 15 ft bls (approximately 5 to 7 feet below the water table), which generally corresponds with the screen interval (6 to 16 ft bls) of the water-table monitoring well network.
- 33 to 35 ft bls (approximately 25 to 27 feet below the water table), which generally corresponds with the screen interval (30 to 40 ft bls) of monitoring well MW-5.
- 58 to 60 ft bls (approximately 50 to 52 feet below the water table) to characterize groundwater quality beneath the existing monitoring well network.

The final groundwater sampling intervals will be determined based on the results of the soil boring program that is designed to determine the three-dimensional extent of NAPL impacts.

The direct push VAP boring groundwater samples will be collected using the Geoprobe® Screen Point Groundwater Sampling System. The assembled Geoprobe® Screen Point Groundwater Sampler will be driven to the target sampling depth. Extension rods will be used to hold the temporary screen in position while the probe rods and sampler sheath are retracted to expose the screen. The sample sheath will be retracted to expose a two-foot screen interval. The sampler sheath will form a mechanical annular seal above the screen interval. Polyethylene tubing will be fitted with a check valve assembly (check valve and check ball) and lowered into the screen interval. The tubing and check valve assembly will be oscillated up and down to pump groundwater to the surface. Once groundwater has been pumped to the surface, the tubing and check valve assembly will be withdrawn from the screen interval and probe rods, and groundwater will be decanted from the tubing to allow for the collection of the groundwater sample. The assembled Geoprobe® Screen Point Groundwater Sampler will then be decontaminated and driven to the next groundwater sampling interval. After the last groundwater sample (58 to 60 ft bls) has been collected, the boring will be grouted from the terminal depth of the boring to land surface.

The sonic boring (SB-7) groundwater samples will be collected using a temporary monitoring well constructed of 2-inch diameter Schedule 40 polyvinyl chloride (PVC) casing and a 2-foot long PVC screen. The temporary monitoring well will be lowered into the drill stem to the target sampling depth. The casing will then be retracted to

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expose the temporary monitoring well screen to the formation. A submersible pump will be lowered into the temporary monitoring well and three well volumes will be purged prior to collecting the groundwater sample. After the groundwater sample has been collected, the temporary monitoring well will be removed from the drill stem and the boring will be advanced to the next groundwater sampling interval. After the target depth of SB-7 has been achieved, the boring will be grouted from the terminal depth of the boring up to the planned depth of the monitoring well and the monitoring well will be installed.

6.2.1.2.2 Groundwater Monitoring Well Installation

Eleven (11) additional monitoring wells (MW-6 through MW-16) will be installed during the RI. The final locations of the monitoring wells may be modified in the field based on the Site reconnaissance and utility locations.

The monitoring wells will be installed at each location using the protocols presented in the ARCADIS *Monitoring Well Installation* SOP (Appendix B [FSP] of this RI Work Plan). The monitoring wells will be constructed using 2-inch diameter Schedule 40 PVC casing and screen. Monitoring wells MW-6 through MW-9 will be completed to a depth that permits the screened section of the well to straddle the water table (anticipated to be 16 feet deep based on the water-table monitoring wells that were installed during the SC). Monitoring wells MW-10 through MW-12 will be completed to a depth that is appropriate to evaluate groundwater quality in the vicinity of the MGP-related NAPL release areas (i.e., screened below the water table). Monitoring wells MW-13 through MW-16 will be completed to a depth that is appropriate to evaluate dissolved-phase impacts outside of the areas where MGP-related NAPL impacts are observed. A 3-foot long sump will be installed at the bottom of monitoring wells that are screened across DNAPL impacts. The monitoring wells will be completed at the surface with a locking cap and a flush-mount protective casing.

Following installation, and immediately prior to development as discussed below, each well will be gauged for the presence of NAPL using the procedures described in the ARCADIS *Water-Level and NAPL Thickness Measurement Procedures* SOP (Appendix B [FSP] of this RI Work Plan). Each well will then be developed by surging and bailing or pumping water from the well using the procedures outlined in the ARCADIS *Monitoring Well Development* SOP (Appendix B [FSP] of this RI Work Plan). Surging and bailing or pumping will continue until the turbidity is below 50 nephelometric turbidity units (NTUs) or until pH and conductivity measurements have stabilized. Water generated by monitoring well development and equipment

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decontamination will be containerized in DOT-approved 55-gallon steel drums or a polyethylene storage tank. Storage vessels will be appropriately labeled with the contents, generator, location, and date for later off-site transportation and disposal by National Grid.

Subsequent to the well installation activities, a New York State licensed surveyor will field survey the monitoring well locations. For each monitoring well, the surveyor will determine the location relative to the New York State Plane Coordinate System, and the ground surface elevation and measuring point elevation (defined as the top of the inner casing) relative to the National Geodetic Vertical Datum of 1929 (NGVD 29).

#### 6.2.1.2.3 Groundwater Flow and Hydraulic Characteristics

Concurrent with the collection of groundwater samples from the direct push temporary monitoring wells (VAP borings), pneumatic slug testing will be conducted at select intervals in select VAP borings to evaluate the hydraulic conductivity of the formation. During pneumatic slug testing, the well head is sealed and air pressure is used to displace/lower the water level. As air pressure in the well is increased, the water level falls until the water pressure "up" and the air pressure "down" are equal. Once the water level is stable, a release valve is quickly opened, instantaneously releasing the air pressure. The water level recovers (rising head test) and a pressure transducer and data logger/computer record the changes in water level and time.

The groundwater flow patterns and hydraulic characteristics beneath the Site will be evaluated by collecting two (2) comprehensive rounds of water-level measurements from the groundwater monitoring wells to determine the groundwater flow direction at the Site. Groundwater levels will be measured to the nearest one-hundredth of a foot from a reference point at the top of the inner casing. The water-level measurements will be converted to groundwater elevations based on the surveyed monitoring well measuring point elevations. The groundwater elevation information will be used to evaluate horizontal groundwater flow beneath the Site, to confirm the hydraulic gradient, and to develop a further understanding of the groundwater flow regime and how it influences contaminant transport.

#### 6.2.1.2.4 Groundwater Quality Characterization

To determine the nature and extent of dissolved-phase MGP-related and/or non-MGPrelated chemical constituents in groundwater at the Site, one complete round of groundwater sampling will be conducted. The groundwater sampling will be conducted

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

two weeks after completion of the monitoring well installation and development activities to allow for a period of equilibration. The groundwater sampling event will consist of collecting one groundwater sample from monitoring wells MW-1 through MW-16. The wells will be purged using low-flow methods as described in the ARCADIS *Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells* SOP (Appendix B [FSP] of this RI Work Plan). Each well will be gauged for the presence of NAPL prior to purging.

Following the purging, one groundwater sample will be collected from each monitoring well using low-flow sampling techniques and a submersible pump. The groundwater samples will be submitted to the laboratory for the analysis of VOCs, SVOCs, TAL metals, and total cyanide. In addition, groundwater samples collected from 3 monitoring wells will also be analyzed for TCL PCBs and TCL pesticides (i.e., 20 percent of the 16 monitoring wells). Field parameters including pH, oxidation-reduction potential (ORP), temperature, conductivity, dissolved oxygen, and turbidity will be collected during groundwater sampling using the procedures outlined in the ARCADIS *Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells* SOP.

#### 6.2.1.2.5 Assess the Presence/Characteristics of NAPL

The monitoring wells will be gauged for the presence of NAPLs during the water-level measurement round. If LNAPL and/or DNAPL are observed to be present in sufficient volume in any monitoring well, the NAPL will be sampled and analyzed for fingerprint analysis, chemical composition, density, viscosity, and interfacial tension. The physical properties analyses will be conducted at ambient groundwater temperatures.

#### 6.2.1.2.6 Management of Investigation-Derived Waste

As described above, IDW will be containerized and staged on site for appropriate characterization and disposal following the procedures outlined in the ARCADIS *Investigation-Derived Waste Handling and Storage* SOP (Appendix B [FSP] of this RI Work Plan). PPE and spent disposable sampling materials will be segregated and placed in DOT-approved 55-gallon steel drums. Decontamination water and monitoring well purge water will be stored in DOT-approved 55-gallon steel drums or a polyethylene storage tank. Waste storage containers will be appropriately labeled with the contents, generator, location, and date for later off-site transportation and disposal by National Grid.

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

One representative sample will be collected from the solid IDW (i.e., drill cuttings) and one representative sample will be collected from the liquid IDW (i.e., development water/monitoring well purge water) generated by the field activities. The samples will be submitted to the laboratory for analysis of the parameters required by the off-site disposal facility. National Grid will use the analytical results from the waste characterization samples to profile the waste materials for disposal.

#### 6.2.1.3 Proposed Soil Vapor Investigation

The proposed soil vapor RI consists of the advancement of four (4) temporary soil vapor points and one (1) ambient air sample at a location upwind of the shopping center. The locations of the proposed temporary soil vapor points are shown on Figure 5. Table 1 provides the complete description/rationale for the proposed scope of work.

The temporary exterior soil vapor points will be advanced to a depth of approximately 5 ft bls using hand excavation soft dig techniques. The temporary soil vapor points will be constructed of stainless steel screens and Teflon®-lined tubing. The temporary soil vapor points will be installed and sampled using the procedures described in the ARCADIS Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation SOP (Appendix B [FSP] of this RI Work Plan).

#### 6.2.2 Data Analysis and Management

Samples will be analyzed in accordance with the analytical methods listed in the QAPP (Appendix C of this RI Work Plan). The chemistry data will be transferred from the laboratory and maintained in a database format. The laboratory will provide Electronic Data Deliverables (EDDs), which will be uploaded directly into the database.

The laboratory will produce NYSDEC ASP Category B deliverable packages and will produce Contract Laboratory Program (CLP)-type data packages that will contain all information needed for formal validation of the data. Data validation will be performed on the data in accordance with analytical method performance criteria, laboratory control limits, NYSDEC ASP Revision 2005 requirements, the USEPA's National Functional Guidelines, and USEPA Region 2 SOPs for data validation. These procedures are specific with regard to evaluation of holding time, surrogate and spike recoveries, precision of duplicate measurements, instrument performance, blank contamination, compound identification, and compound quantification. Data will be qualified as necessary in accordance with the SOPs. Additional information is



# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

provided in the QAPP (Appendix C of this RI Work Plan). Following completion of the above validation, data usability summary reports (DUSRs) will be prepared in accordance with DER-10 and appended to the RI Report.

#### 6.2.3 Remedial Investigation Deliverables

Following full evaluation and analysis of the field and analytical data, a determination will be made as to the validity of the CSM. If it is determined that additional characterization is necessary, those investigative activities will be detailed in a letter submitted to the NYSDEC, as described in Section 5.1 of this RI Work Plan. The comprehensive RI Report will be submitted following completion of the RI and a full evaluation of the RI data; the submittal of the RI Report and subsequent approval by the NYSDEC will conclude the RI process.

#### 6.2.4 Sampling and Analysis Plan

The SAP is the umbrella document that consists of Appendices A through C of this RI Work Plan. The SAP includes the following elements:

- The DNAPL Contingency Plan (Appendix A) describes detailed procedures to be followed during drilling to limit the potential for remobilization and downward migration of DNAPL.
- The FSP (Appendix B) defines field sampling and data collection methods and procedures consistent with NYSDEC DER-10 (NYSDEC, 2010).
- The QAPP (Appendix C) describes the QA/QC protocols necessary to achieve the data quality objectives.

Additional plans that are part of the RI Work Plan include the following:

The CAMP (Appendix D) describes air monitoring and response efforts to provide a measure of protection for the downwind community during performance of the RI consistent with NYSDEC DER-10 (NYSDEC, 2010).

The HASP (Appendix E) presents the site-specific procedures to protect Site workers conducting the RI field activities.

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

The CPP (Appendix F) was developed in accordance with NYSDEC DER-23 *Citizen Participation Handbook for Remedial Programs* (NYSDEC, 2010).

#### 6.2.5 Evaluation of Data Gaps and Refining RI Objectives

During the course of the data collection and evaluation tasks described in previous sections, remaining or new data gaps may be identified. If clear and pertinent data gaps are identified, they will be addressed with the NYSDEC with the goal of limiting the interruption in the field work.

#### 6.2.6 Human Health Exposure Assessment

Upon completion of data collection and analysis, a qualitative human health exposure assessment (QHHEA) will be performed and an exposure assessment report will be prepared and included with the RI Report. The QHHEA will be conducted in accordance with NYSDEC DER-10 (NYSDEC, 2010). The purpose of the QHHEA is to determine whether Site conditions pose an unacceptable hazard to potentially exposed receptor populations. To pose an unacceptable hazard to receptor populations, the receptor must be exposed to contaminants at the Site. The QHHEA will evaluate whether complete exposure pathways exist at the Site and identify constituents of potential concern (COPCs) for those receptors and media of concern where a complete exposure pathway exists (NYSDEC, 2010).

#### 6.2.7 Remedial Investigation Report

Once sufficient information is collected to complete the RI and address the RI objectives, as described in Section 1.1 of this RI Work Plan, the RI Report will be prepared and submitted to the NYSDEC for review. The RI Report will incorporate the SC data generated prior to the RI, will be prepared following applicable NYSDEC guidance, and will be consistent with the requirements of the Order on Consent for the Site.

#### 7. Project Management Plan

For project responsibilities and communication see the organization chart in the QAPP (Appendix C of this RI Work Plan). Subcontractors used for specialty services, such as drilling, laboratory analysis, and surveying will be subcontractors that ARCADIS has relied on for similar tasks performed previously. Any subcontractor utilized will meet the requirements of the NYSDEC.

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

#### 7.1 Project Schedule

The schedule for implementing the RI activities presented in this RI Work Plan is provided as Figure 6. The project duration will depend on whether additional investigation efforts are required, as described in Section 5.1 of this RI Work Plan.

The NYSDEC will be provided with five (5) days advance notice of the commencement of field work. In general, the sequence of field activities and related rationale for the RI are as follows:

- 1. Conduct pre-field planning including field verification of sampling locations and utility mark-outs.
- 2. Perform RI field work. The RI data will provide information on whether further characterization of COCs is needed.
- If necessary based on the data collected in item 2, prepare a letter detailing additional activities that may be required, as described in Section 5.1 of this RI Work Plan.
- 4. Prepare and issue RI Report.

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

#### 8. References

- ARCADIS. 2009. Final Site Characterization Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York, Site No. 224047, Index # A2-0552-0606. April 2009.
- ARCADIS. 2010. Site Characterization Work Plan Addendum Vapor Intrusion Investigation, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York, Site No. 224047, Index # A2-0552-0606. February 2010.
- ARCADIS. 2010a. Site Characterization Data Summary, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York, Site No. 224047, Index # A2-0552-0606. April 2010.
- ARCADIS. 2010b. Site Characterization Data Summary Addendum, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York, Site No. 224047, Index # A2-0552-0606. May 2010.
- Environmental Data Resources, Inc. 2008. Aerial Photo Decade Package from 1954 2006, July 2008.
- GEI Consultants, Inc. 2007. Dangman Park Former MGP Site, Site Number 224047, Records Search. April 2007.
- McClymonds, N.E. and Franke, O.L. 1972. Water-Transmitting Properties of Aquifers on Long Island, New York. United States Geological Survey Professional Paper 627-E.
- New York City Planning Commission Zoning Map. July 29, 2009.
- New York State Department of Environmental Conservation. 2010. DER-10 Technical Guidance for Site Investigation and Remediation. May 2010.
- New York State Department of Environmental Conservation. 2010. DER-23 Citizen Participation Handbook for Remedial Programs. January 2010.
- New York State Department of Health. 2006. Guidance for Evaluating Soil Vapor Intrusion in the State of New York. October 2006.

# Remedial Investigation Work Plan

Former Dangman Park Manufactured Gas Plant Site

Smolensky, D.A., Buxton, H.T., and Shernoff, P.K. 1989. Hydrologic Framework of Long Island, New York. U.S. Geological Survey Hydrologic Investigations Atlas HA-709.

#### Table 1. Summary of Proposed Remedial Investigation Activities and Sampling Rationale, Former Dangman Park MGP Site, Brooklyn, New York.

Sample Location	Sample Method	Sample Sequence	Rationale	Proposed Total Depth (ft bls)	Samples Collected <sup>1, 2</sup>	
Soil Borings/Monitoring Wells						
SB-4/MW-10	Sonic Drilling with 4-inch Diameter Core Barrel Sampling	1	<ul> <li>Vertically delineate NAPL impacts observed in SB-3</li> <li>Boring will extend to a depth 10 ft below impacted soil</li> <li>The monitoring well will be screened to evaluate groundwater quality below the NAPL impacts</li> </ul>	80	<ul> <li>1 soil sample collected at a depth 10 feet impacted soil to aid in vertical delineation</li> <li>1 soil sample collected for physical prope analyses from a depth interval where the ge degree of impacts is observed</li> </ul>	
SB-5/MW-11	Sonic Drilling with 4-inch Diameter Core Barrel Sampling	2	<ul> <li>Vertically delineate NAPL impacts observed in SB-1</li> <li>Boring will extend to a depth 10 ft below impacted soil</li> <li>The monitoring well will be screened across the greatest apparent degree of NAPL impacts to evaluate groundwater quality and recover drainable NAPL (if present)</li> </ul>	80	<ul> <li>1 soil sample collected at a depth 10 feet impacted soil to aid in vertical delineation</li> <li>1 soil sample collected for physical prope analyses from a depth interval where the gr degree of impacts is observed</li> </ul>	
SB-7/MW-12	Sonic Drilling with 4-inch Diameter Core Barrel Sampling and PVC Temporary Monitoring Well	4	<ul> <li>Evaluate soil and groundwater quality on Lot 25 and delineate impacts observed in SB-1 and SB-2</li> <li>The monitoring well will be screened across the same interval as MW-11</li> </ul>	80	<ul> <li>1 soil sample collected from 2 - 3 ft bls</li> <li>If no impacts are observed then 1 soil sat collected just above the water table</li> <li>If impacts are observed then 1 soil sampl from the depth interval where the greatest of impacts is observed</li> <li>3 groundwater samples collected from ter monitoring well</li> </ul>	
<u>Soil Borings</u>						
SB-6	Sonic Drilling with 4-inch Diameter Core Barrel Sampling	3	• Evaluate soil quality on Lot 25 and delineate impacts observed in SB-1 and SB-2	80	<ul> <li>1 soil sample collected from 2 - 3 ft bls</li> <li>If no impacts are observed then 1 soil sat collected just above the water table</li> <li>If impacts are observed then 1 soil sampl from the depth interval where the greatest of impacts is observed</li> </ul>	
SB-8 through SB-15	DP with Dual Tube Sampling	5	Evaluate soil quality and delineate impacts observed during Site Characterization	80	<ul> <li>1 soil sample collected from 2 - 3 ft bls</li> <li>If no impacts are observed then 1 soil sat collected just above the water table</li> <li>If impacts are observed then 1 soil sampl from the depth interval where the greatest of impacts is observed</li> </ul>	
SB-16 through SB-23 (Step Out Soil Borings, if necessary)	DP with Dual Tube Sampling	6	Evaluate soil quality and delineate impacts	80	<ul> <li>1 soil sample collected from 2 - 3 ft bls</li> <li>If no impacts are observed then 1 soil san collected just above the water table</li> <li>If impacts are observed then 1 soil sampl from the depth interval where the greatest of impacts is observed</li> </ul>	

See footnotes on last page.

#### Sample Analysis <sup>3</sup>

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1 Soil Sample Analyzed for: VOCs, SVOCs, TAL Metals, TCL PCBs, Total Cyanide, Free Cyanide 1 Soil Sample Analyzed for: Physical Properties Analyses

2-3 Soil Samples Analyzed for: VOCs, SVOCs, TAL Metals, TCL PCBs, Total Cyanide, Free Cyanide <sup>4</sup> 3 Groundwater Samples Analyzed for: VOCs, SVOCs

2-3 Soil Samples Analyzed for: VOCs, SVOCs, TAL Metals, TCL PCBs, Total Cyanide, Free Cyanide <sup>4</sup>

2-3 Soil Samples Analyzed for: VOCs, SVOCs, TAL Metals, TCL PCBs, Total Cyanide, Free Cyanide <sup>4</sup>

2-3 Soil Samples Analyzed for: VOCs, SVOCs, TAL Metals, TCL PCBs, Total Cyanide, Free Cyanide <sup>4</sup>

Sample Location	Sample Method	Sample Sequence	Rationale	Proposed Total Depth (ft bls)	Samples Collected <sup>1, 2</sup>
Temporary Monitoring Wells					
VP-1 through VP-5	DP Screen Point Groundwater Sampler	7	Determine the nature and distribution of dissolved-phase groundwater impacts	60	3 groundwater samples collected from ea monitoring well
Temporary Soil Vapor Points					
SV-1 through SV-4	Temporary Soil Vapor Point	8	<ul> <li>Evaluate shallow soil vapor quality on Lots 1 and 25</li> </ul>	5	1 soil vapor sample collected from 4.5 to each temporary soil vapor point
1 2 3 4 DP VOCs SVOCs PCBs	One round of groundwater samples wil	Il be collected from groundwater sam r Compound and	•	I for VOCs, SVOCs, TAL met	als, and Total Cyanide.
TAL TCL ft bls	Target Analyte List. Target Compound List. Feet below land surface.				

#### Table 1. Summary of Proposed Remedial Investigation Activities and Sampling Rationale, Former Dangman Park MGP Site, Brooklyn, New York.

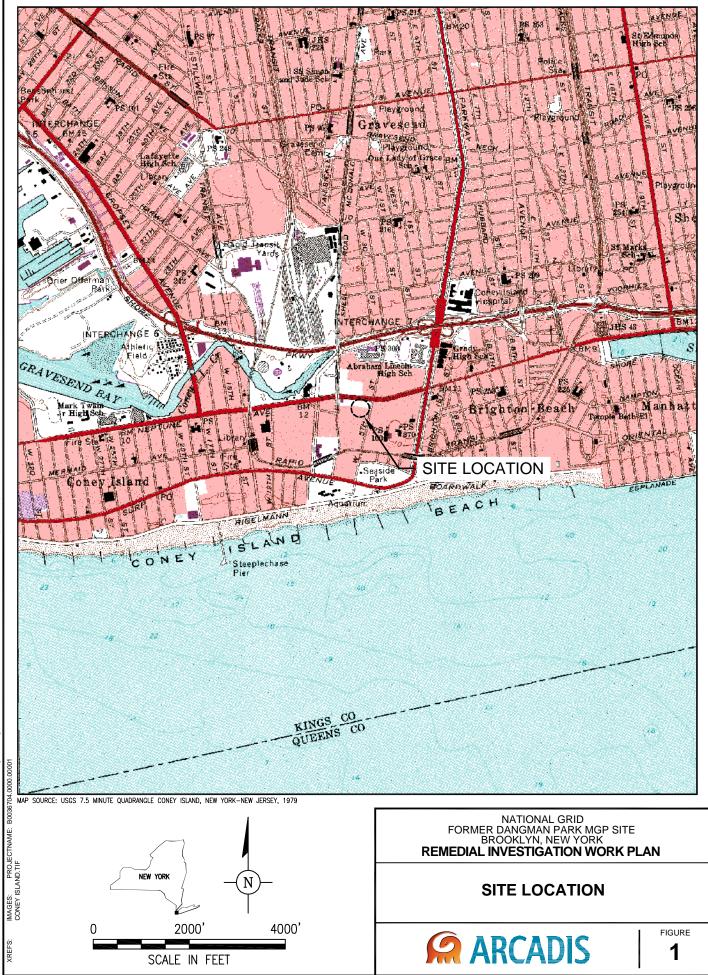
#### Sample Analysis <sup>3</sup>

m each temporary

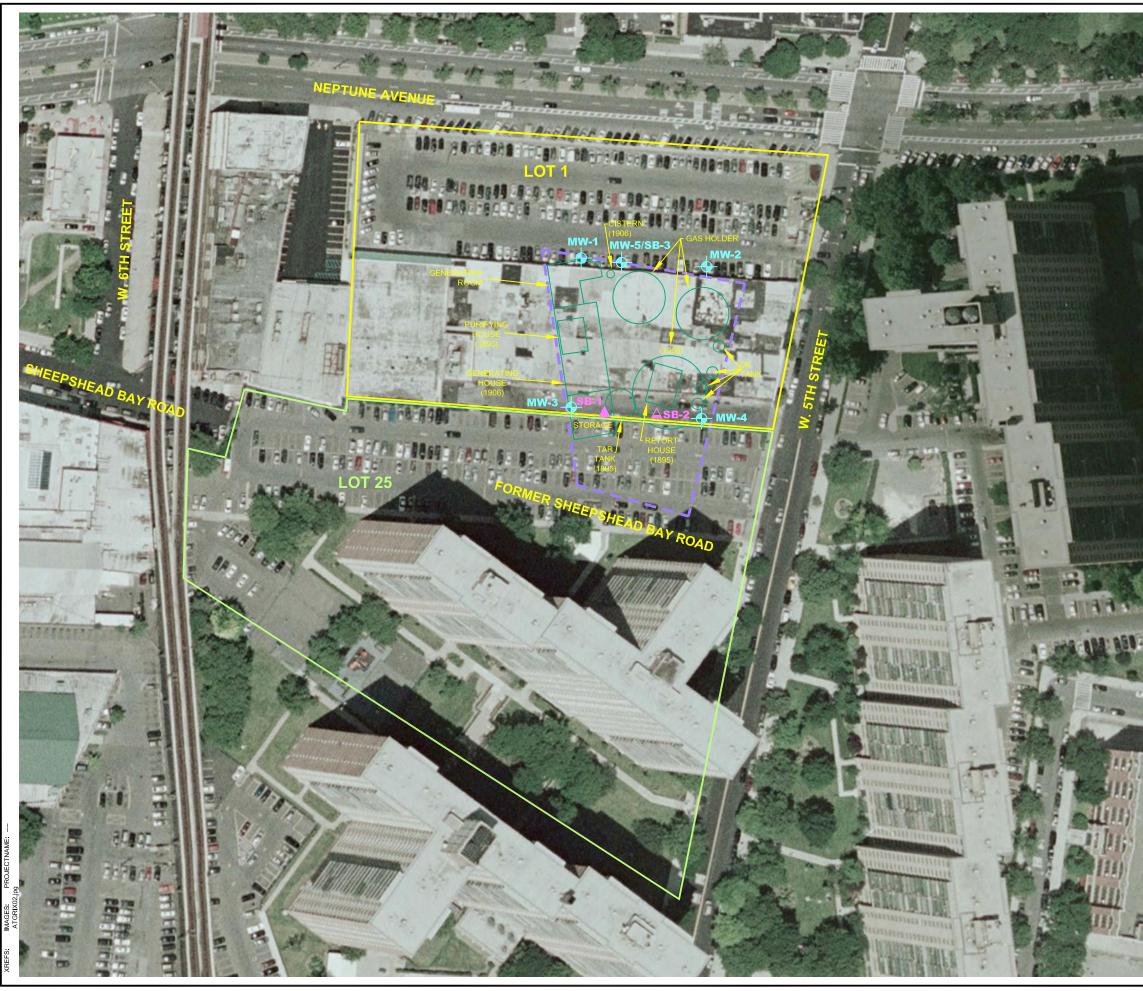
3 Groundwater Samples Analyzed for: VOCs, SVOCs

.5 to 5 ft bls in

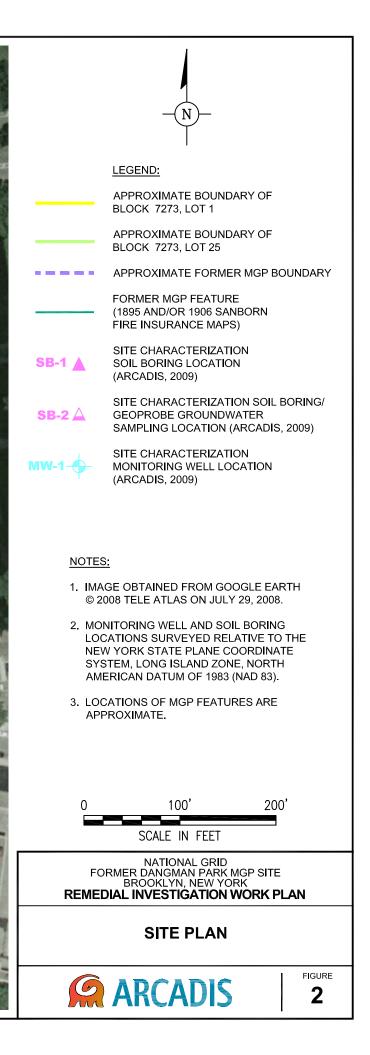
1 Soil Vapor Sample Analyzed for: TO-15 VOCs Expanded

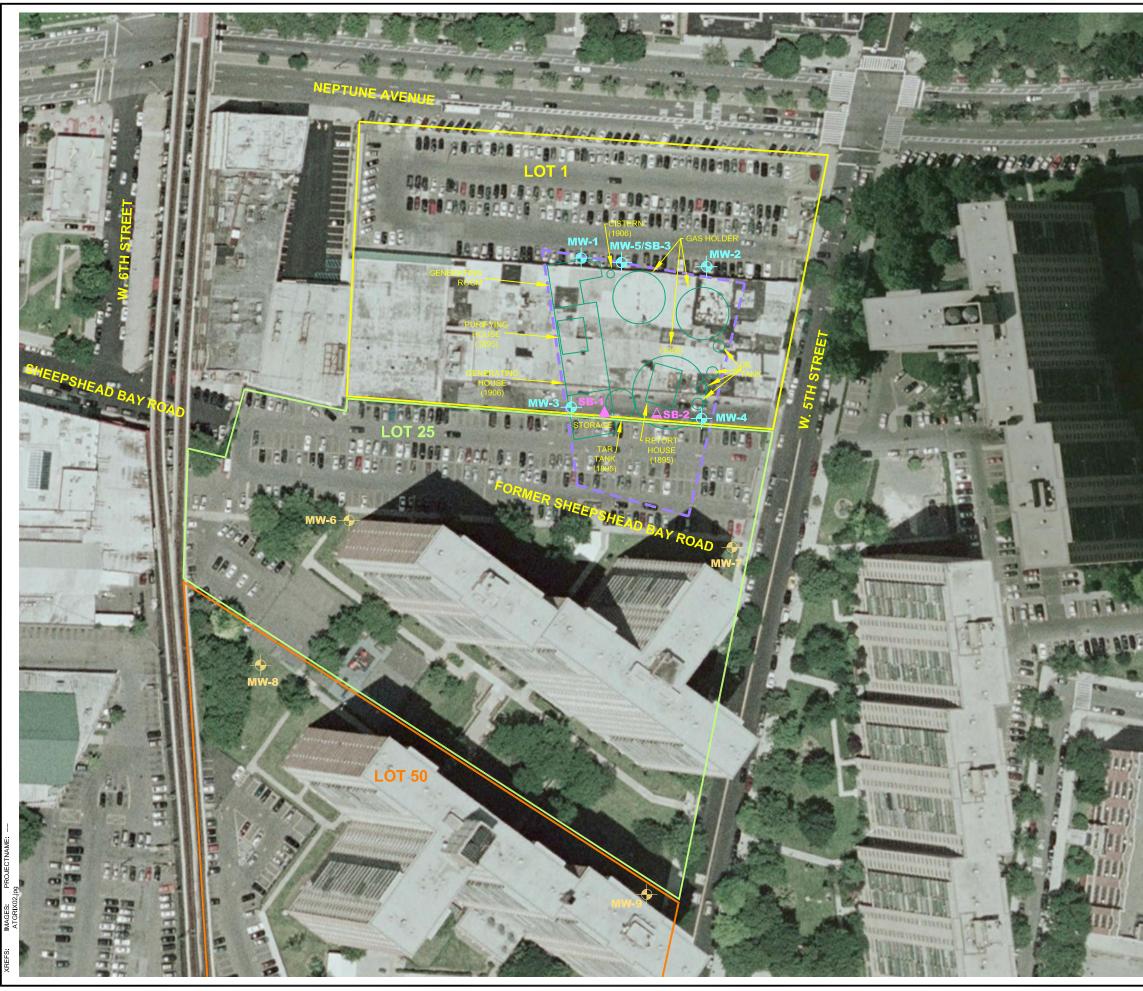


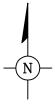
SANCHEZ, ADRIAN 8/27/2010 7:56 PM BY: ARCADIS\_MELVILLE.CTB PLOTTED: PDFPLOTSTYLETABLE: 17.1S (LMS TECH) PAGESETUP: 8/27/2010 7:56 PM ACADVER: LYR:ON=\*;OFF=\*REF\* UT: 1SAVED: 8/27 TM:CK LYR J LAYOUT: Dwb. PM:SF CITY:MELVILLE-NY DIV/GROUP:ENR1 DB:ALS LD: PIC:JN PM: G:/ENVCADMelville-NYACT/B0036704(0001/00001/RI Work Plan/Figure 7



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LEGEND:

APPROXIMATE BOUNDARY OF BLOCK 7273, LOT 1

APPROXIMATE BOUNDARY OF BLOCK 7273, LOT 25

APPROXIMATE BOUNDARY OF BLOCK 7273, LOT 50

FORMER MGP FEATURE (1895 AND/OR 1906 SANBORN FIRE INSURANCE MAPS)

SB-1



SITE CHARACTERIZATION SOIL BORING LOCATION (ARCADIS, 2009) SITE CHARACTERIZATION SOIL BORING/

GEOPROBE GROUNDWATER SAMPLING LOCATION (ARCADIS, 2009)



MW-6 🔶

SITE CHARACTERIZATION MONITORING WELL LOCATION (ARCADIS, 2009)

PROPOSED WATER-TABLE MONITORING WELL

#### NOTES:

1. IMAGE OBTAINED FROM GOOGLE EARTH © 2008 TELE ATLAS ON JULY 29, 2008.

2. MONITORING WELL AND SOIL BORING LOCATIONS SURVEYED RELATIVE TO THE NEW YORK STATE PLANE COORDINATE SYSTEM, LONG ISLAND ZONE, NORTH AMERICAN DATUM OF 1983 (NAD 83).

3. PROPOSED MONITORING WELL LOCATIONS WILL BE ADJUSTED IN THE FIELD AS NEEDED BASED ON ACCESSIBILITY AND UTILITY CLEARANCE.

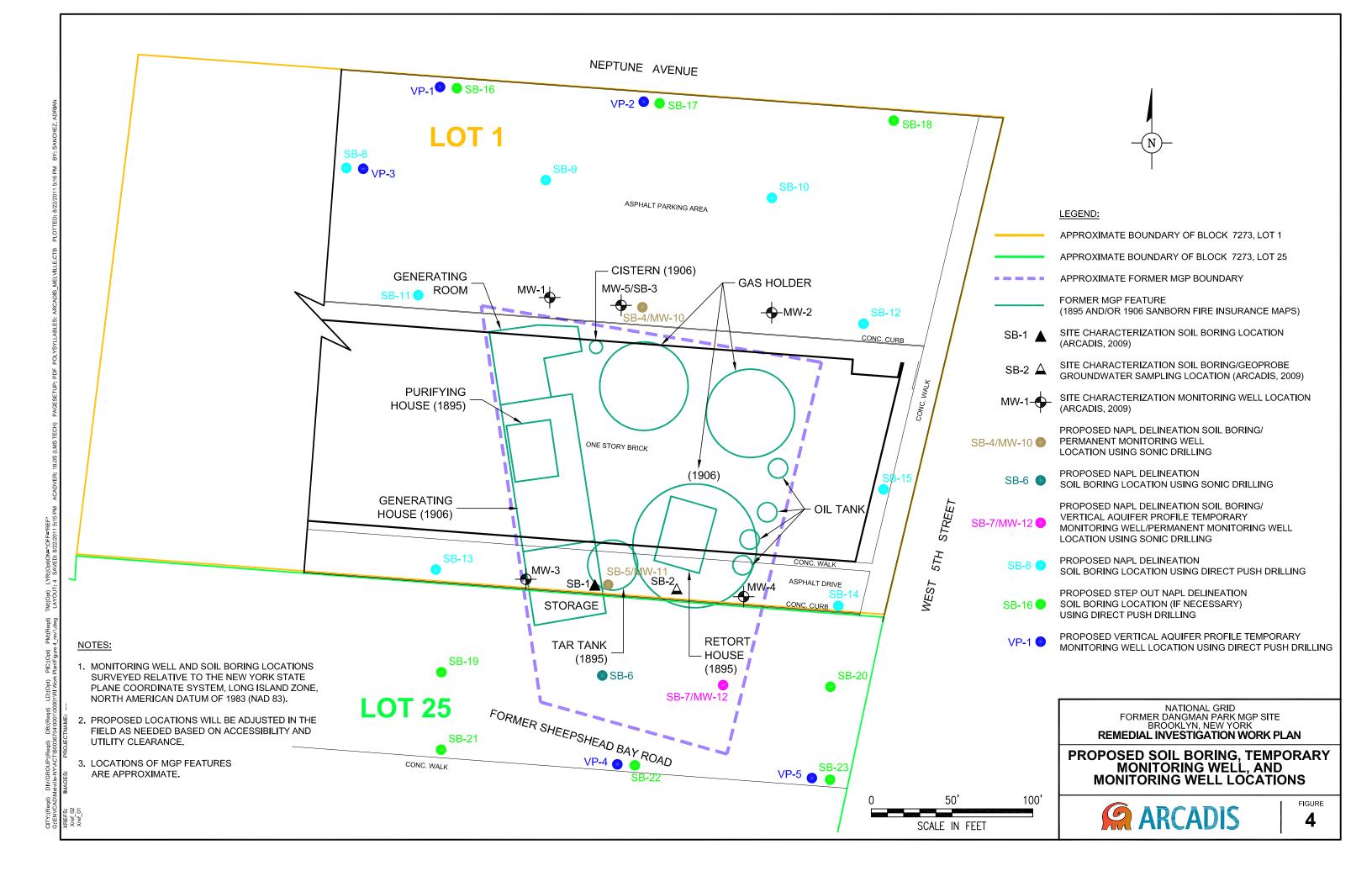
4. LOCATIONS OF MGP FEATURES ARE APPROXIMATE.

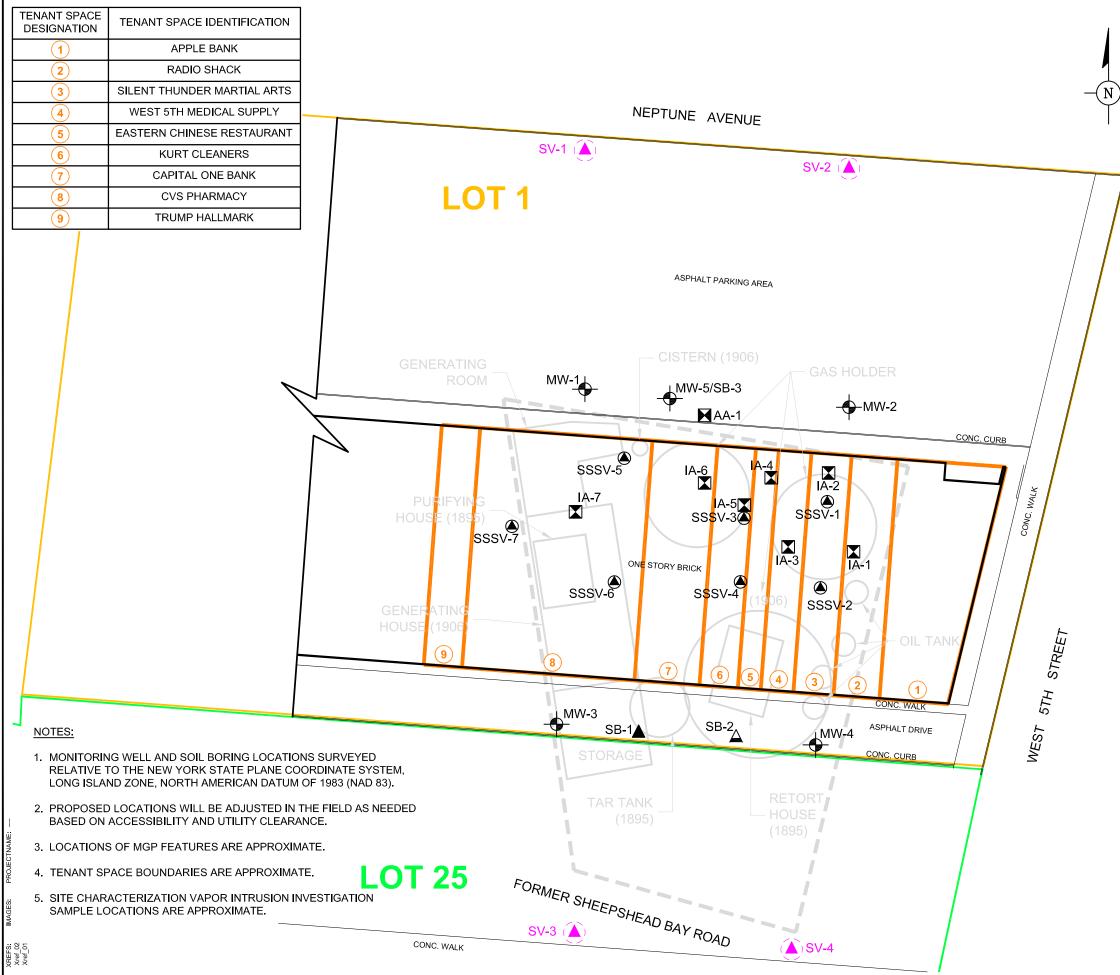
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	SCALE IN	FEET	

NATIONAL GRID FORMER DANGMAN PARK MGP SITE BROOKLYN, NEW YORK **REMEDIAL INVESTIGATION WORK PLAN** 

#### PROPOSED WATER-TABLE MONITORING WELL LOCATIONS







	LEGEND:
_	APPROXIMATE BOUNDARY OF BLOCK 7273, LOT 1
	APPROXIMATE BOUNDARY OF BLOCK 7273, LOT 25
	APPROXIMATE FORMER MGP BOUNDARY
	FORMER MGP FEATURE (1895 AND/OR 1906 SANBORN FIRE INSURANCE MAPS)
	TENANT SPACE BOUNDARY
(	1 TENANT SPACE DESIGNATION
SB-1	SITE CHARACTERIZATION SOIL BORING LOCATION (ARCADIS, 2009)
SB-2	SITE CHARACTERIZATION SOIL BORING/ GEOPROBE GROUNDWATER SAMPLING LOCATION (ARCADIS, 2009)
MW-1 <del>-(</del>	SITE CHARACTERIZATION MONITORING WELL LOCATION (ARCADIS, 2009)
SSSV-1 (	<ul> <li>SITE CHARACTERIZATION SUB-SLAB</li> <li>SOIL VAPOR SAMPLE LOCATION (ARCADIS, 2010)</li> </ul>
IA-1 [	SITE CHARACTERIZATION INDOOR AIR QUALITY SAMPLE LOCATION (ARCADIS, 2010)
AA-1	SITE CHARACTERIZATION AMBIENT AIR QUALITY SAMPLE LOCATION (ARCADIS, 2010)
SV-1 🤅	PROPOSED SOIL VAPOR POINT LOCATION
	0 50' 100' SCALE IN FEET
	NATIONAL GRID FORMER DANGMAN PARK MGP SITE BROOKLYN, NEW YORK <b>REMEDIAL INVESTIGATION WORK PLAN</b>
	PROPOSED SOIL VAPOR POINT LOCATIONS
	ARCADIS

ID Task Name		Duration	Start	Finish	1	Sep '11	Oct '11	Nov '11	Dec '11	Jan '1	2 Feb '12	Mar '12		May '12
						11 18 25 2	9 16 23 30		4 11 18 25	1 8 15 22			1 8 15 22 29	6 13 20 2
1	Submit Final Remedial Investigation Work Plan to NYSDEC	1 day	Fri 9/2/11	Fri 9/2/11	le 🏟 🏟	I	I	1	I		I	I	I I	
					1	1	1	1	1		1		I I	
3	Remedial Investigation Implementation	103 days	Wed 9/7/11	Fri 1/27/12		I	1	1	1		I I	1	I I I	
4	Phase 1 (Water-Table Monitoring Well Installation)	5 days	Wed 9/7/11	Tue 9/13/11	; 👝		1		і І			1		
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5	Collect Water-Level Measurements from Water-Table Monitoring Wells	1 day	Mon 9/19/11	Mon 9/19/11	1		1	1	I		I I	1	I I	
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6	Phase 2 (Source Area NAPL Delineation)	30 days	Mon 9/26/11	Fri 11/4/11	1			1	1		1	1	I I I I	
7	Phase 3 (Vertical Aquifer Profile Sampling)	10 days	Mon 11/7/11	Fri 11/18/11		1	1	<u> </u>	!		1		I I	
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8	Phase 4 (Deep Monitoring Well Installation)	10 days	Mon 12/12/11	Fri 12/23/11		1	1				1		I I	
0	Collect Water-Level Measurements from Entire Monitoring Well Network	5 days	Mon 1/9/12	Fri 1/13/12	1	1	I I		1	+	I I	1	I I	
9	Collect Water-Level Measurements from Entire Monitoring Weil Network	5 uays	WOIT 1/9/12	FIL 1/13/12	1	1	1	1	1		1		I I	
10	Groundwater Sampling	5 days	Mon 1/16/12	Fri 1/20/12	1	1	I I		1	<b>*</b>	1	I I	I I	
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11	Soil Vapor Sampling	5 days	Mon 1/23/12	Fri 1/27/12		1	I		1	· · · · · · · · · · · · · · · · · · ·	1			
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12	Remedial Investigation Report	60 days	Mon 3/5/12	Fri 5/25/12		1	1	1	1		1		· · ·	
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Project: Dangman Park MGP Site Date: Fri 8/19/11	Task Split	( <u></u> )	Progress Milestone	<b>♦</b>	Summary Project Summary	External Tasks	Deadline	Ŷ
\\arcadis-us.com\OfficeData\Melville-NY\.	APROJECT\Nationa	al Grid\Dangman Park\RI W	ork Plan\Final RI V	Vork Plan\RI Project Sche	dule_Figure 6.mpp			

### Appendix A

DNAPL Contingency Plan



Imagine the result

### **DNAPL Contingency Plan**

Rev. #: 3

Rev Date: May 1, 2010

#### **Approval Signatures**

David Lipson Prepared by:

Date: 5/1/10

Reviewed by: Mu

Michael Gefell (Technical Expert)

Date: 5/1/10

#### I. Scope and Application

This document has been prepared to guide drilling activities at sites where there is a reasonable expectation that dense, non-aqueous phase liquid (DNAPL) may be present, and provide procedures to be implemented in the event that DNAPL is encountered during subsurface investigations. These procedures are proposed to limit the potential of remobilizing DNAPL, if any, in response to drilling and sampling activities. In addition, the procedures are designed to optimize the recovery of encountered DNAPL (if any) in a safe and efficient manner. This DNAPL Contingency Plan was developed based on a similar document prepared by DNAPL expert Bernard H. Kueper, Ph.D., P.Eng., of Queens University, for an EPA Region 1 Superfund Site (Kueper, May 1995).

Downward DNAPL mobilization may occur in response to drilling activities (shortcircuiting along drill stem and/or completed well screen) and groundwater extraction (creation of downward hydraulic gradient in excess of previously measured downward gradients). This DNAPL Contingency Plan addresses drilling-related issues.

#### II. Personnel Qualifications

DNAPL contingency field activities will be performed by persons who have been trained in proper drilling and well installation procedures under the guidance of an experienced field geologist, engineer, or technician.

#### III. Equipment List

The following materials will be available during soil boring and monitoring well installation activities, as required:

- Work Plan, Field Sampling Plan (FSP), and site Health and Safety Plan (HASP);
- personal protective equipment (PPE), as required by the HASP;
- equipment specified under drilling and well installation SOPs;
- photo-ionization detector (PID) or flame ionization detector (FID)
- hydrophobic dye (Oil Red O or Sudan IV), pertinent at chlorinated solvent sites;
- disposable pans for performing soil-water pan tests; and
- clean, empty jars for performing soil-water shake tests.

• field notebooks and/or personal digital assistant (PDA)

#### IV. Cautions

#### Downward Mobilization

DNAPL can migrate downward during drilling and well installation processes, or via the sand pack or screen of a monitoring well. This caution is applicable to all DNAPL sites, but may be especially important at solvent sites, where DNAPL is likely to have relatively high density and low viscosity; also, pure solvents may be clear and colorless, and therefore difficult to detect visually. Other DNAPLs such as coal tar and creosote, or waste solvents that have been used in degreasing operations, commonly have a dark color and are readily visible in soil or water samples.

#### Direct-Push Drilling

DNAPL can be indirectly detected in the soil using direct-push instruments such as TarGost (which detects coal tar or creosote DNAPL due to fluorescence) or membrane interface probe (also known as MIP, which detects volatile organic compounds). These types of devices can also be used with a cone-penetration tool (CPT) to rapidly characterize stratigraphy in addition to potential DNAPL presence. However, currentlyavailable direct push tools do not allow the borehole to be grouted as the direct-push tool is extracted from the subsurface. Therefore, use of direct-push tools within zones that are known or likely to contain DNAPL should be limited to one or more the following situations: 1) each direct-push boring is terminated at the first indication of potential DNAPL; 2) direct-push is used for lateral DNAPL delineation above a widespread, thick capillary barrier that has been previously characterized using standard soil borings; 3) a direct-push tool is advanced inside of an outer casing, such that the boring can be tremie-grouted from the bottom upward during removal of the outer casing. Wherever possible, direct-push detectors (e.g., MIP or TarGOST) should be "calibrated" in terms of their response using a series of standards prepared using site soil and DNAPL - this process can significantly improve the reliability of interpretations regarding DNAPL presence.

#### **Other Considerations**

The presence or absence of DNAPL at a site can have significant implications in terms of site management, health and safety, and the feasibility of potential remedial alternatives. Therefore, field personnel must be attentive to the potential for DNAPL, recognize when DNAPL is encountered during drilling, and accurately document field observations indicating the presence of DNAPL and interpreted DNAPL depth. In addition, opportunities to characterize DNAPL, when present, may be rare. When

practicable, DNAPL samples should be collected and analyzed for physical and chemical characteristics.

#### Shipping

A Shipping Determination must be performed, by DOT-trained personnel, for all environmental and geotechnical samples that are to be shipped, as well as some types of environmental equipment/supplies that are to be shipped.

#### V. Health and Safety Considerations

Field activities associated with this DNAPL Contingency Plan will be performed in accordance with the site HASP, a copy of which will be present on site during such activities.

#### VI. Procedure

#### **DNAPL Screening During Overburden Drilling**

To screen for the potential presence of DNAPL in soil, drilling procedures must allow for high-quality porous media samples to be taken. Split-spoon samples or direct-push samplers should be taken continuously in 2-foot intervals ahead of the auger or drill casing. Upon opening each split-spoon sampler or direct-push plastic liner sleeve, the soil will immediately be evaluated for the presence of visible non-aqueous phase liquid (NAPL), screened for the presence of organic vapors using a portable photo-ionization detector (PID) or flame ionization detector (FID). During screening, the soil will be split open using a clean spatula or knife and the PID or FID probe will be placed in the opening and covered with a gloved hand. Such readings will be obtained along the entire length of the sample. If NAPL is immediately visible in the sample, its depth should be noted and the sampling team should skip to the fourth bullet below.

If the PID or FID examination reveals the presence of organic vapors above 100 parts per million (ppm), the sample will undergo further detailed evaluation for visible non-aqueous phase liquid (NAPL). The assessment for NAPL will include a combination of the following tests/observations:

 Evaluation for Visible NAPL Sheen or Free-Phase NAPL in Soil Sampler – The NAPL sheen will be a colorful iridescent appearance on the soil sample. NAPL may also appear as droplets or continuous accumulations of liquid with a color typically ranging from yellow to brown to black, depending on the type of NAPL. Creosote DNAPL (associated with wood-treating sites) and coal-tar DNAPL (associated with manufactured gas plant [MGP] sites) are typically black and

have a characteristic, pungent odor. Pure chlorinated solvents may be colorless in the absence of hydrophobic dye. Solvents mixed with oils may appear brown.

- Soil-Water Pan Test A portion of the selected soil interval with the highest PID or FID reading > 100 ppm will be placed in a disposable polyethylene dish along with a small volume of potable or distilled water. The dish will be gently tilted back and forth to mix the soil and water, and the surface of the water will be viewed in natural light to observe the development of a sheen, if any. A small quantity of Oil Red O or Sudan IV hydrophobic dye powder will be added and the soil and dye will be manually mixed for approximately 30 to 60 seconds and smeared in the dish to create a paste-like consistency using a new nitrile glove-covered hand. A positive test result will be indicated by a sheen on the surface of the water and/or a bright red color imparted to the soil following mixing with dye.
- Soil-Water Shake Test A small quantity of soil (up to 15 cc) will be placed in a clear, colorless, jar containing an equal volume of potable or distilled water (40-mL vials are well suited to this purpose, but not required). After the soil settles into the water, the surface of the water will be evaluated for a visible sheen under natural light. The jar will be closed and gently shaken for approximately 10 to 20 seconds. Again, the surface of the water will be evaluated for a visible sheen or a temporary layer of foam. A small quantity (approximately 0.5 to 1 cc) of Oil Red O or Sudan IV powder will be placed in the jar. The sheen layer, if present, will be evaluated for a reaction to the dye (change to bright red color). The jar will be closed and gently shaken for approximately 10 to 20 seconds. The contents in the closed jar will be examined under natural light for visible bright red dyed liquid inside the jar. A positive test result will be indicated by the presence of a visible sheen or foam on the surface of water, a reaction between the dye and the sheen layer upon first addition of the dye powder, a bright red coating on the inside of the vial (particularly above the water line), or red-dyed droplets within the soil.
- Estimation of Relative Degree of NAPL Saturation When NAPL is interpreted as present in a particular portion of soil, the field geologist should attempt to estimate the relative degree of NAPL saturation in the soil. Specifically, based on the apparent, visible continuity of NAPL within the soil, an interpretation should be made as to whether the observed NAPL is: apparently pooled (continuous interval of soil across entire diameter of soil sample in which the pore spaces are filled with a mixture of NAPL and water); apparently residual (isolated droplets or blebs of NAPL, surrounded by pore spaces containing only water); or inconclusive (unclear whether pooled or residual). If NAPL freely drains out of a soil sample, that indicates that the NAPL is in the form of a pool however, pooled NAPL may not always freely drain out of soil samples.

As mentioned previously, if NAPL is obviously present upon opening the soil sampler or evaluating the soil sample within the split-spoon sampler or direct-push liner sleeve, it is not necessary to perform a soil-water pan test or soil-water shake test. In addition, it is not necessary to perform both a soil-water pan test and a soil-water shake test; either test method is acceptable. The pan test may be preferred in some circumstances because the presence of a sheen may be easier to see on a wider surface.

When using hydrophobic dye in the tests above, color will be assessed outdoors under natural light during the period between sunrise and sunset, regardless of the degree of cloud cover.

The results of each test or observation will be recorded in the field notebook and/or PDA.

#### **DNAPL Screening During Bedrock Drilling**

To screen for the potential presence of DNAPL in bedrock, drilling fluids, rock cuttings, and/or core samples are monitored for the presence of sheens. During drilling using rotary methods (coring or roller bit drilling with water or drilling mud), the return fluid will be screened with a PID or FID and evaluated continuously for the presence of a sheen in the recirculation tub. Where core samples are obtained, they will be carefully evaluated for the presence of a sheen on fracture surfaces. During drilling using airrotary methods, rock cuttings will be continuously screened using a PID or FID and evaluated for the presence of a sheen. During drilling with rotary methods, the positive head level at the borehole will reduce the potential for DNAPL short-circuiting via the borehole.

If a sheen is observed with any of these methods, drilling will be temporarily discontinued and an evaluation will be undertaken to determine whether pooled DNAPL is present. The drill stem will be retracted to a few feet above the apparent depth where the sheen was first encountered. Groundwater will be extracted from the borehole to produce a drawdown of approximately 5 feet below the approximate static, non-pumping water level for a period of 20 minutes to test for the presence of pooled, mobilizable DNAPL in the fractures surrounding the open borehole. The bottom of the borehole will then be evaluated for the presence of DNAPL using an interface probe or bottom-loading bailer. If no DNAPL is observed, the interpretation will be made that the sheen was not produced by pooled DNAPL. In this case, if drilling by the rotary method, the recirculation water will be replaced by clean water and drilling will continue. Replacing the recirculation water reduces the potential for cross-contamination and facilitates observation of a newly created sheen, if any, at a deeper interval. Accumulation of DNAPL in the bottom of the borehole, however, indicates

that the boring has encountered pooled DNAPL. If DNAPL has accumulated, it will be removed using a bottom-loading bailer or pump.

#### Data Collection Below Zone Containing Pooled DNAPL

If pooled DNAPL is encountered in a borehole and deeper drilling is required to collect data below the zone containing pooled DNAPL, one of the following actions will be taken.

- 1. <u>Adjustment of Drilling Location</u> The boring where pooled DNAPL was encountered will be abandoned by tremie grouting using neat cement grout and a replacement boring will be re-attempted at a nearby location.
- 2. <u>DNAPL Sump Installation</u> A DNAPL collection well will be installed with a blank sump properly grouted in place below the screen and the boring will be reattempted at a nearby location. In this case, after removing the DNAPL in the borehole, the boring may be advanced an additional 1 to 2 feet to accommodate a blank sump below the interval with apparent pooled DNAPL.
- 3. Casing Off DNAPL Layers - If pooled DNAPL is found to be present throughout an area where deeper drilling is essential, a permanent, grouted casing should be installed. The bottom of the pooled DNAPL likely coincides with the top of a relatively fine-grained, low permeability, stratum (capillary barrier). Permanent casing will be installed to the bottom of the borehole and grouted in place using the displacement method prior to advancing the borehole any further. Via the displacement method (also known as the Halliburton Method or the packerinjection method), grout is displaced out the bottom of the casing and fills the annulus outside the casing from the bottom upward. In this case, after removing any DNAPL that may have accumulated in the borehole, the boring may be advanced a few feet into the top of the underlying confining layer or up to 5 feet in bedrock prior to grouting the casing to assist in isolating the zone containing apparently pooled DNAPL. The bottom of the borehole should be checked for the DNAPL accumulation prior to installing and grouting the casing in the drilled "socket". When the casing is grouted in place and the grout has set, the drilling recirculation water will be replaced with clean water to prevent crosscontamination and facilitate observation of a newly created sheen (if any) at a deeper interval, and drilling will continue.

#### **DNAPL Monitoring**

New wells installed in borings where DNAPL was encountered during drilling will be monitored for DNAPL accumulation in the DNAPL sump using an oil-water interface

probe or bottom-loading bailer within approximately one day following initial installation. If DNAPL is encountered, a bottom-loading bailer or pump will be used to remove the DNAPL, the final DNAPL thickness will be recorded, and the DNAPL thickness will be reassessed after another day of accumulation (if any). This process will be repeated until DNAPL no longer accumulates overnight, at which point the accumulation monitoring and removal period will extend to one-week intervals. If no DNAPL accumulation is observed over a period of one week, further DNAPL monitoring may be continued with a longer period between monitoring events.

Any DNAPL recovered during drilling and monitoring activities should be analyzed for chemical composition, DNAPL-water interfacial tension, density, and viscosity. The physical tests should be performed at the approximate groundwater temperature at the site where the DNAPL sample was obtained, typically between 10°C and 20°C. These parameters will allow for correlation of groundwater chemistry with suspected DNAPL locations and will allow an estimate to be made of the volume and potential mobility of DNAPL, if any, in the formation.

#### VII. Waste Management

DNAPL removed from wells will be temporarily stored on-site in metal drums for subsequent appropriate off-site disposal. The locations and volumes of recovered DNAPL will be noted.

#### VIII. Data Recording and Management

Any occurrence of DNAPL encountered during subsurface investigations will be documented in an appropriate field notebook in terms of the drilling location (boring or well identification), depth below surface, type of geologic material in which DNAPL was observed, field screening and testing results, and apparent degree of DNAPL saturation (pooled or residual), and visual characteristics of DNAPL (e.g., color or qualitative viscosity). DNAPL locations and depths will be recorded in a field book and/or on subsurface log forms, as appropriate.

#### IX. Quality Assurance

DNAPL can be mobilized downward as a result of drilling operations. It is very difficult to drill through DNAPL without bringing about vertical DNAPL mobilization. This opinion is stated by USEPA (1992): "In DNAPL zones, drilling should generally be minimized and should be suspended when a potential trapping layer is first encountered. Drilling through DNAPL zones into deeper stratigraphic units should be avoided." The DNAPL screening procedure outlined in this plan should, therefore, be implemented while drilling at all locations and depths within overburden or bedrock

where potential DNAPL presence is suspected. If data collection is required below a zone containing DNAPL, the interval containing DNAPL will be cased off prior to drilling deeper.

#### X. References

Kueper, B.H., May 11, 1995. DNAPL Contingency Plan. [Prepared at the request of *de maximis, inc.*].

United States Environmental Protection Agency (USEPA), 1992. Memorandum from D. Clay: Considerations in Ground-Water Remediation at Superfund Sites and RCRA Facilities – Update. OSWER Directive No. 9283.1-06.

### Appendix B

Field Sampling Plan



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### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

March 7, 2011

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#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

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#### 9. Waste Management and Disposal

#### Attachments

- B-1 Standard Operating Procedures
- B-2 National Grid Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites

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#### Appendix B Field Sampling Plan

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#### 1. Introduction

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this Field Sampling Plan (FSP) as a component of the Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York. The FSP describes the methods and procedures to be used for environmental sample collection during implementation of the RI field activities.

The FSP should be used in conjunction with the RI Work Plan, the DNAPL Contingency Plan, the Quality Assurance Project Plan (QAPP), the Community Air Monitoring Plan (CAMP), and the Health and Safety Plan (HASP). The RI Work Plan presents the Site background and defines the field sampling program. The DNAPL Contingency Plan describes detailed procedures to be followed during drilling to limit the potential for remobilization and downward migration of dense non-aqueous phase liquid (DNAPL). The QAPP presents the quality assurance/quality control (QA/QC) procedures to be used during implementation of the RI Work Plan, as well as a description of the general field and laboratory procedures. The CAMP provides procedures to protect the downwind communities from potential airborne releases of constituents of concern during RI activities. The DNAPL Contingency Plan, QAPP, CAMP, and HASP are provided as Appendices A, C, D, and E of the RI Work Plan, respectively.

#### 1.1 Plan Organization

This FSP contains the following sections:

- Section 2: Site Description
- Section 3: "Remedial Investigation Activities" summarizes the type of sampling to be performed in accordance with the FSP
- Section 4: "Pre-Field Preparation and Equipment" describes preparation and equipment needed prior to mobilization to the field
- Section 5: "Remedial Investigation Field Activities" describes the sampling and data collection associated with the following RI activities:
  - o Drilling of soil borings and collection of soil samples

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- Drilling of temporary monitoring wells and collection of groundwater samples
- o Drilling and installation of monitoring wells
- Collection of hydraulic (water-level and fluid-level) measurements from monitoring wells
- o Collection of groundwater samples from monitoring wells
- o Drilling of temporary soil vapor points and collection of soil vapor samples
- o Collection of ambient (outdoor) air quality samples
- Section 6: Investigation-Derived Waste (IDW) Sampling
- Section 7: Sample Collection, Labeling, Handling, and Analysis
- Section 8: Field Decontamination Procedures
- Section 9: Waste Management and Disposal

#### 2. Site Description

The Site is located at 486 Neptune Avenue in the Borough of Brooklyn, New York City, Kings County, New York and occupies portions of two parcels that are identified by Tax Map Number: Block 7273, Lots 1 and 25. As shown on Figure 1 of the RI Work Plan, the Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is generally flat with an elevation of approximately 9 feet above mean sea level (msl). The closest natural surface water body is Coney Island Creek, which is located approximately 0.25 miles to the northwest of the Site.

The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5<sup>th</sup> Street to the east, a residential parcel to the south, and a commercial parcel to the west. Currently, the Site is developed with a shopping center and a parking lot for a high-rise apartment building.

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#### 3. Remedial Investigation Activities

The primary objectives of the proposed RI activities are listed in Section 1.1 of the RI Work Plan. Sample collection efforts include obtaining discrete soil samples from soil borings, groundwater samples from temporary and permanent monitoring wells, soil vapor samples from temporary soil vapor points, ambient air quality samples, and IDW solid and liquid media samples for waste characterization purposes.

#### 4. Pre-Field Preparation and Equipment

Pre-field preparation will be performed prior to mobilization to the field to conduct the activities described in the RI Work Plan and this FSP. The field project team (scientists, technicians, and engineers) will be responsible for obtaining, operating, and maintaining the required equipment, for procuring and maintaining sample containers or canisters pertinent to the collection of environmental samples, and collecting the samples as specified herein. The equipment and materials required to perform the RI field activities are listed in the standard operating procedures (SOPs) provided in Attachment B-1 of this FSP.

In general, the pre-cleaned environmental sample containers (bottles) or canisters (SUMMA® canisters) will be provided by the analytical laboratory in accordance with procedures and requirements set forth in the QAPP (Appendix C of the RI Work Plan). The sample containers or canisters will be inventoried and inspected prior to sampling to verify that the required containers or canisters are present and in good condition.

#### 5. Remedial Investigation Field Activities

The following sections describe the sampling methods associated with the RI activities.

#### 5.1 Sample Locations

The locations of the proposed RI soil borings, temporary and permanent monitoring wells, and temporary soil vapor points are shown on Figures 3 through 5 of the RI Work Plan.

Underground and aboveground utilities will be identified prior to any drilling or subsurface sampling. Utilities will be located by contacting responsible agencies by phone (New York's DigNet of New York City & Long Island) so that their underground utilities can be marked at the Site. Utilities will be located and identified following the

#### Appendix B Field Sampling Plan

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procedures outlined in the ARCADIS *Utility Location Policy and Procedure* (see HASP). Other potential on site hazards such as traffic and building hazards will be identified during a Site reconnaissance visit. The work will be planned, in conjunction with Site occupants, to minimize impacts and promote safe conditions for workers, Site occupants, and visitors.

#### 5.2 Soil Borings

This section describes the methods to drill soil borings and collect soil samples. The soil borings will be drilled using direct push or sonic drilling methods (see RI Work Plan for details).

If a soil boring will be drilled using direct push drilling techniques, the Geoprobe® Dual Tube Sampling System will be used. The Geoprobe® Dual Tube Sampling System employs an outer casing and inner rod string assembly. Soil sampling will be conducted at the boring locations by advancing 3.25-inch outside diameter (OD) probe rods (outer casing) and a 5-foot long sample liner. Dual tube sampling uses two sets of probe rods to collect continuous soil cores. One set of rods is driven into the ground as an outer casing. These rods receive the driving force from the hammer and provide a sealed borehole from which soil samples may be collected. The second, smaller set of rods (inner rod string) are placed inside the outer casing. The inner rod string hold the sample liner in place as the outer casing is driven over the sampling interval. The inner rod string is then retracted to retrieve the filled sample liner.

If using sonic drilling techniques, soil sampling will be conducted at the boring locations by driving a 5-foot long 4-inch diameter core barrel using vibration, rotation, and a downward force. Once the core barrel has been advanced, a 6-inch diameter secondary or "override" casing will be advanced down to the same depth as the inner core barrel. The override casing keeps the borehole from collapsing while the inner core barrel is removed. Once the core barrel is removed, the soil core will be extruded from the core barrel.

Soil recovered from each sample interval will be visually characterized for color, texture, and moisture content as described in the National Grid *Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites* (Attachment B-2 of this FSP). The presence of visible staining, non-aqueous phase liquid (NAPL), and obvious odors will be noted. If NAPL is encountered in any of the soil borings, the DNAPL Contingency Plan provided in Appendix A of the RI Work Plan will be



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implemented to limit the potential for remobilization and downward migration of DNAPL.

Soil samples will be collected following the procedures described in the ARCADIS *Soil Drilling and Sample Collection* SOP (Attachment B-1 of this FSP) and as described in the RI Work Plan and Section 7.1 of this FSP. Soil samples will be analyzed for the compounds/analytes specified in the RI Work Plan and the QAPP (Appendix C of the RI Work Plan).

The drilling and sampling of soil borings will include the following activities:

- 1. Determine location of the soil boring and avoid aboveground and underground utilities per the HASP.
- 2. The approximate location will be measured and shown on a location sketch.
- 3. The drill rig will be mobilized to the proposed location.
- 4. The proposed location will be cleared of utilities to a depth of approximately 5 feet below land surface (ft bls) by hand digging or by air knife.
- Drilling will commence and soil cores will be collected as specified in the RI Work Plan.
- 6. The field geologist (in coordination with Driller) will monitor the formation drilled through evaluation of collected soil cores.
- 7. Soil samples will be collected for the specified analyses (see RI Work Plan and QAPP for details).
- 8. Boreholes will be abandoned as described in Section 6.2.1.1 of the RI Work Plan.

#### 5.3 Temporary Monitoring Wells

This section describes the methods to collect groundwater samples from temporary monitoring wells. The temporary monitoring wells will be drilled using direct push or sonic drilling methods (see RI Work Plan for details).

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The temporary monitoring wells (VP-1 through VP-5) groundwater samples will be collected using the Geoprobe® Screen Point Groundwater Sampling System. The assembled Geoprobe® Screen Point Groundwater Sampler will be driven to the target sampling depth. Extension rods will be used to hold the temporary screen in position while the probe rods and sampler sheath are retracted to expose the screen. The sample sheath will be retracted to expose a two-foot screen interval. The sampler sheath will form a mechanical annular seal above the screen interval. Polyethylene tubing will be fitted with a check valve assembly (check valve and check ball) and lowered into the screen interval. The tubing and check valve assembly will be oscillated up and down to pump groundwater to the surface. Once groundwater has been pumped to the surface, the tubing and check valve assembly will be withdrawn from the screen interval and probe rods, and groundwater will be decanted from the tubing to allow for the collection of the groundwater sample. The assembled Geoprobe® Screen Point Groundwater Sampler will then be decontaminated and driven to the next groundwater sampling interval. After the last groundwater sample has been collected, the boring will be grouted from the terminal depth of the boring to land surface.

The sonic boring (SB-7) groundwater samples will be collected using a temporary monitoring well constructed of 2-inch diameter Schedule 40 polyvinyl chloride (PVC) casing and a 2-foot long PVC screen. The temporary monitoring well will be lowered into the drill stem to the target sampling depth. The casing will then be retracted to expose the temporary monitoring well screen to the formation. A submersible pump will be lowered into the temporary monitoring well and three well volumes will be purged prior to collecting the groundwater sample. After the groundwater sample has been collected, the temporary monitoring well will be removed from the drill stem and the boring will be advanced to the next groundwater sampling interval. After the target depth of SB-7 has been achieved, the boring will be grouted from the terminal depth of the boring up to the planned depth of the monitoring well and the permanent monitoring well will be installed.

#### 5.4 Monitoring Well Drilling, Installation, and Development

This section describes the methods to drill, install, and develop monitoring wells. The monitoring wells will be drilled using hollow-stem auger or sonic drilling methods. Based on available information on the local geology, bedrock is expected to be present at a depth greater than 100 ft bls; thus, bedrock is not expected to be encountered at the Site. After completion of drilling and well installation, the monitoring wells will be developed to establish hydraulic connection between the well and the formation. The

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monitoring wells will be installed at each location using the procedures described in the ARCADIS *Monitoring Well Installation* SOP (Attachment B-1 of this FSP).

#### 5.4.1 Drilling

The drilling and geological logging methods to be completed in connection with monitoring well installation are as follows:

- The MW-6 through MW-9 (water-table monitoring wells) boreholes will be drilled using hollow-stem auger methods. Soil borings will be completed to a depth as discussed in the RI Work Plan. Continuous soil cores will be collected in the MW-6 through MW-9 monitoring well borings using a 2-foot long, 2-inch outside diameter (OD) split-spoon sampler.
- The MW-10 through MW-16 boreholes will be drilled using sonic methods. Soil borings will be completed to a depth as discussed in the RI Work Plan. Continuous soil cores will be collected in the MW-10 through MW-16 monitoring well borings using a 5-foot long 4-inch diameter core barrel.
- The designated field geologist will log borehole geology (using the Unified Soil Classification System [ASTM D2488]) and monitoring well specifications in the field book and/or field forms.
- Soil cuttings will be placed in drums supplied by the drilling subcontractor. Decontamination water will be placed in drums supplied by the drilling subcontractor or a polyethylene tank supplied by National Grid. Soil cuttings and decontamination water will be containerized at the end of each work day. The open-top drums used to contain IDW will be covered when not in use.

#### 5.4.2 Monitoring Well Specifications

The monitoring wells will be installed according to the following specifications:

- 2-inch diameter threaded, flush-joint Schedule 40 polyvinyl chloride (PVC) casing and 10-foot long, 20 slot (0.020-inch) screens will be installed.
- A sump, 3 feet in length and <u>sealed in with neat cement grout</u>, may be attached to the bottom of the screen for potential collection of DNAPL, if present (or suspected).

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- The annular space around the screens will be backfilled with a Morie #1 (or equivalent) sand pack to a height of 1 to 2 feet above the top of the screen.
- A 2-foot thick bentonite seal (pellets or slurry depending on depth of seal) will be placed above the sand pack. The bentonite seal must be allowed to hydrate before placing grout above the seal.
- The remainder of the annular space will be filled with a cement/bentonite grout to approximately 2 ft bls. The grout will be placed from the bottom up using a tremie pipe. The grout will consist of a cement mixture of one 94-pound bag of Portland cement, approximately 5 pounds of granular bentonite, and approximately 7 gallons of water. The grout will be allowed to set for a minimum of 48 hours before wells are developed if the grout is placed below the water table.
- Each monitoring well will be completed with an 8-inch diameter water-tight flush-mount protective casing. A 2-foot by 2-foot cement pad will be installed around the flush-mount protective casing. The well casing will extend to within approximately 2 to 4 inches below land surface and will include a locking cap.
- The north side of the top of the PVC well casing will be marked and the elevation surveyed to the nearest 0.01 foot.
- The measuring point on wells will be the marked location on the innermost PVC casing.

The following characteristics of each newly installed monitoring well will be recorded in the field log book and/or on a Well Construction Log:

- Date/time of construction
- Drilling method and drilling fluid (if used)
- Approximate well location
- Borehole diameter and well casing diameter
- Well depth

#### Appendix B Field Sampling Plan

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- Casing materials
- Screen materials and design
- Casing and screen joint type
- Screen slot size/length
- Filter pack material/size
- Filter pack placement method
- Sealant materials
- Sealant placement method
- Surface seal design/construction
- Well development procedure
- Type of protective well cap
- Detailed drawing of well (including dimensions)

If saturated conditions are encountered just above a confining layer, then a monitoring well may be installed to the top of the confining layer. If a sump is being installed to monitor for the potential presence of DNAPL, the sump should be installed such that the top of the bentonite and sump are at or slightly below the top of the confining layer.

The on-site geologist shall specify the monitoring well design to the drilling contractor before installation. An alternate monitoring well construction method may be used if the water table is within approximately 4 feet of land surface. If these conditions are encountered, the thickness of the sand pack and bentonite seal would be reduced as necessary and the depth of the protective casing would be modified as necessary.

#### 5.4.3 Monitoring Well Development

A minimum of 48 hours after installation, or the day after installation if the grout is located above the water table, the monitoring wells will be developed by surging and

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

pumping using the procedures outlined in the ARCADIS *Monitoring Well Development* SOP (Attachment B-1 of this FSP). The development water will be containerized in drums to be provided by the drilling subcontractor or in a polyethylene tank provided by National Grid. The wells will be developed until the turbidity is reduced to 50 nephelometric turbidity units (NTUs) or less or until the pH and conductivity measurements have stabilized. Following development, the monitoring wells will be allowed to equilibrate for a minimum of two weeks before groundwater sampling is conducted. Monitoring well development will be overseen by a qualified person and the duration, method of development, and approximate volume of water removed will be recorded in the field book.

#### 5.5 Hydraulic Measurements in Monitoring Wells

Hydraulic (i.e., water-level and fluid-level) measurements will be collected using the procedures described in the ARCADIS *Water-Level and NAPL Thickness Measurement Procedures* SOP (Attachment B-1 of this FSP). The measurements will be made in as short a timeframe as practical to minimize temporal fluctuations in hydraulic conditions.

#### 5.6 Monitoring Well Groundwater Sample Collection

Groundwater sampling will be conducted using the procedures described in the ARCADIS *Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells* SOP (Attachment B-1 of this FSP). Groundwater samples will be analyzed for the compounds/analytes specified in the RI Work Plan and the QAPP (Appendix C of the RI Work Plan).

#### 5.7 Temporary Soil Vapor Points

The temporary soil vapor points will be installed and sampled using the procedures described in the ARCADIS *Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation* SOP (Attachment B-1 of this FSP). The temporary soil vapor point boreholes will be advanced to a depth of approximately 5 ft bls using hand excavation soft dig techniques. The temporary soil vapor point samples are intended to serve as screening-level samples that will be collected from a temporary point; therefore, temporal repeat sampling of temporary soil vapor point sample intervals will not be performed. Soil vapor samples will be analyzed for the compounds specified in the RI Work Plan and the QAPP (Appendix C of the RI Work Plan).

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

#### 5.8 Ambient Air Quality Sample Collection

Ambient air quality sampling will be conducted using the procedures described in the ARCADIS *Ambient Air Sampling and Analysis Using USEPA Method TO-15* SOP (Attachment B-1 of this FSP). Ambient air quality samples will be analyzed for the compounds specified in the RI Work Plan and the QAPP (Appendix C of the RI Work Plan).

#### 6. Investigation-Derived Waste Sampling

In general, IDW will be containerized in DOT-approved 55-gallon steel drums. Solid and liquid samples will be collected to support IDW characterization for disposal purposes. Solid IDW samples will be collected from the 55-gallon drums by opening the drums, collecting a number of grab samples using a stainless steel spoon/trowel, compositing the grab samples in a stainless steel bowl (except samples collected for VOC analysis), and transferring the sample into the sample containers. The VOC sample will be immediately transferred directly into the laboratory-supplied sample bottles. Liquid/water IDW samples will be collected from the 55-gallon drums by opening the drum, collecting a grab sample using a bailer, and decanting the sample directly into the sample containers. IDW samples will be analyzed by the laboratory for the parameters specified by the receiving/disposal facility.

#### 7. Sample Collection, Labeling, Handling, and Analysis

Samples (including QA/QC samples specified in the QAPP) will be properly labeled and identified, and the applicable sampling log and Chain-of-Custody Form will be completed. The QAPP provides additional details regarding Field Records and QA/QC samples, frequency and protocols, sample labeling, and sample custody. Sample containers and SUMMA® canisters will be checked for proper identification/labeling and compared to the Chain-of-Custody Form for accuracy prior to packaging any sample for shipment. The Chain-of-Custody Form will be placed in a sealed plastic bag and taped to the underside of the cooler lid (soil and groundwater samples) or placed in the canister shipping box (air samples). The samples will be handled, packed, and shipped using the procedures described in the ARCADIS *Chain-of-Custody, Handling, Packing and Shipping* SOP (Attachment B-1 of this FSP).

The soil and groundwater samples will be wrapped with a cushioning material to preclude sample container breakage during shipment and placed in a cooler. Sufficient amounts of bagged ice will be placed in the cooler to keep the samples at 4

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

degrees Celsius until arrival at the laboratory. The cooler will be sealed with packaging tape and custody seals will be placed in such a manner that any opening of the cooler prior to arrival at the laboratory can be visually detected.

The canister (air samples) shipping box will be sealed with packaging tape and custody seals will be placed in such a manner that any opening of the box prior to arrival at the laboratory can be visually detected.

Samples will be delivered by overnight carrier or laboratory courier to the analytical laboratory following sample custody requirements specified in the QAPP. The laboratory will be prepared to receive the samples and perform preliminary extractions or analyses within the analytical method recommended holding times.

#### 7.1 Soil Samples

Soil sampling to be conducted as part of the RI includes the collection of soil samples from soil borings (see RI Work Plan for details). Soil samples will be collected using the procedures described in the ARCADIS *Soil Drilling and Sample Collection* SOP (Attachment B-1 of this FSP) and as described in this FSP and the RI Work Plan. Soil samples will be collected from the appropriate two-foot interval using a decontaminated stainless steel spoon or trowel. The VOC sample will be immediately transferred directly into the laboratory-supplied sample bottles. A sufficient amount of the remaining soil will be homogenized by mixing the sample in a decontaminated stainless steel bowl with a decontaminated stainless steel spoon or trowel using the procedures described in the ARCADIS *Compositing or Homogenizing Samples* SOP (Attachment B-1 of this FSP). Laboratory-supplied sample containers for other analytes (i.e., SVOCs, total cyanide, and free cyanide) will then be filled. All sample bottle caps will be secured snugly, but not over-tightened.

The soil cores will be screened for organic vapors using the jar headspace method and a PID following the procedures outlined in the ARCADIS *Photoionization Detector Air Monitoring and Field Screening* SOP (Attachment B-1 of this FSP). In addition, a geologist will be on site during the drilling operations to describe each sample in accordance with the National Grid *Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites* (Attachment B-2 of this FSP), and will include:

- Depth
- Sample recovery

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

- Soil type and sorting
- Color (natural color, discoloring due to impacts should be described as staining)
- Moisture content (dry, moist, wet)
- Texture
- Grain size and shape
- Relative density
- Consistency (degree of plasticity for fine-grained soils; viscosity for NAPL such as taffy-like, etc.)
- Representativeness
- Visible evidence of residues (petroleum-like sheen, tar-like material, oil-like material, ash-like material, etc.; also describe distribution and percentage of area impacted)
- Miscellaneous observation (obvious odors e.g., faint odor, moderate odor, strong odor, etc.)

#### 7.2 Groundwater Samples

Groundwater samples will be collected directly into the laboratory-supplied sample bottles. The flow of water from the sampling equipment will be adjusted to ensure slow, laminar flow so that no entrained air bubbles are present in VOC samples. Special care will be taken in filling and capping volatile organic analysis (VOA) vials so that headspace/air bubbles are not present in the groundwater samples. In addition, overflowing bottles will be avoided to prevent the loss of floating substances or preservatives that may have already been added to the bottle. All sample bottle caps will be secured snugly, but not over-tightened.

The groundwater samples will be described in accordance with the National Grid *Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites* (Attachment B-2 of this FSP), and will include any visual observations of impacts, odors, or coating

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

of sampling equipment. This information will be recorded on the Water Sampling Log or Low-Flow Groundwater Sampling Log.

#### 7.3 Soil Vapor and Ambient Air Quality Samples

Soil vapor samples and ambient air quality samples will be collected directly into the laboratory-supplied SUMMA® canisters. Sample time duration will be monitored during sampling and the SUMMA® canister valve will be closed when the vacuum is 5 inches of Hg.

#### 8. Field Decontamination Procedures

Decontamination procedures for non-dedicated field equipment are presented in detail in this section and include decontamination procedures associated with non-dedicated sampling equipment and downhole drilling tools and equipment. In general, after decontamination is completed, items will be stored in a manner to preserve their decontaminated condition prior to use.

#### 8.1 Drilling Equipment Decontamination

A decontamination pad will be constructed, lined with plastic sheeting, and will contain a sump for water collection. The sump will be lined with plastic and be of sufficient volume to accommodate the decontamination water generation needs. Drilling equipment including the rear-end of the drilling rig (if necessary), augers, bits, drill rods, tools, and tremie pipe will be cleaned on the decontamination pad with a high-pressure hot water "steam cleaner" unit and scrubbed with a wire brush, as needed, to remove foreign material (e.g., soil, tar, and oil). The equipment will be decontaminated prior to the start of drilling activities, between each borehole, and prior to leaving the Site. Tools, drill rods, and augers will be placed on sawhorses, decontaminated pallets, or polyethylene plastic sheets following steam cleaning. Direct contact with the ground will be avoided. Decontamination water will be containerized in DOT-approved 55gallon open-top steel drums or a polyethylene tank. Open-top drums will remain closed when not in use.

Following decontamination of Site equipment, the decontamination pad will be decommissioned. The decommissioning will be completed by:

#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

- Transferring the bulk of the remaining liquids and solids into the drums or tanks to be provided by the drilling subcontractor or National Grid for these materials.
- Rolling the sheeting used in the decontamination pad onto itself to prevent discharge of the remaining materials to the ground surface. Once rolled up, the polyethylene sheeting will be placed in the drums used for disposal of personal protective equipment (PPE) and disposable equipment.

#### 8.2 Sampling Equipment Decontamination

Soil sampling and groundwater sampling equipment requiring decontamination includes, but is not limited to, split-spoon samplers, core barrels, stainless steel spoons/trowels/bowls, interface meters, and non-dedicated pumps/appurtenances. The sampling equipment will be decontaminated following the procedures described in the ARCADIS *Field Equipment Decontamination* SOP (Attachment B-1 of this FSP). Water quality probes and water-level indicators will be decontaminated by rinsing with distilled water. Decontamination may be conducted at the sampling location as long as liquids are contained in pails or buckets. The equipment will be decontaminated before and between each use and prior to demobilization. At no time will washed equipment be placed directly on the ground. Decontaminated equipment will either be used immediately or wrapped in plastic or aluminum foil for storage or transportation from the designated decontamination area to the sampling location. Decontamination fluids will be containerized prior to off-site transportation and disposal as described in Section 9 of this FSP.

#### 9. Waste Management and Disposal

Solid and liquid IDW generated during field activities including, but not limited to, drill cuttings, monitoring well purge water, and decontamination water will be managed and disposed as outlined in this section. IDW will be containerized in appropriate waste containers and staged on site prior to off-site disposal following the procedures described in the ARCADIS *Investigation-Derived Waste Handling and Storage* SOP (Attachment B-1 of this FSP). If the property owners do not permit the IDW to be stored on site, then the IDW will be picked up on a daily basis and transported to a treatment, storage, and disposal facility (TSDF) for temporary storage until the waste has been characterized and profiled for acceptance at the off-site disposal facility. Soil cuttings, PPE, and spent disposable sampling materials will be segregated by waste type and placed in Department of Transportation (DOT)-approved 55-gallon steel



#### Appendix B Field Sampling Plan

Former Dangman Park Manufactured Gas Plant Site

drums. Decontamination water and development water will be stored in DOTapproved 55-gallon steel drums or a polyethylene storage tank. Storage vessels will be appropriately labeled with the contents, generator, location, and date for later offsite transportation and disposal by National Grid.



#### Attachment B-1

Standard Operating Procedures



Imagine the result

# Soil Drilling and Sample Collection

Rev. #: 1

Rev Date: March 3, 2009

**Approval Signatures** 

anon Prepared by:

Date: 3/3/09

Johner Reviewed by: (Technical Expert)

Date: 3/3/09

#### I. Scope and Application

Overburden drilling is commonly performed using the hollow-stem auger drilling method. Other drilling methods suitable for overburden drilling, which are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, Rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary. Direct-push techniques (e.g., Geoprobe or cone penetrometer) may also be used. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling depths, site or regional geologic knowledge, types of sampling to be conducted, required sample quality and volume, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools).

#### II. Personnel Qualifications

The Project Manager (a qualified geologist, environmental scientist, or engineer) will identify the appropriate soil boring locations, depth and soil sample intervals in a written plan.

Personnel responsible for overseeing drilling operations must have at least 16 hours of prior training overseeing drilling activities with an experienced geologist, environmental scientist, or engineer with at least 2 years of prior experience.

#### III. Equipment List

The following materials will be available during soil boring and sampling activities, as required:

- Site Plan with proposed soil boring/well locations;
- Work Plan and site Health and Safety Plan (HASP);
- personal protective equipment (PPE), as required by the HASP;
- drilling equipment required by the American Society for Testing and Materials (ASTM) D 1586, when performing split-spoon sampling;
- disposable plastic liners, when drilling with direct-push equipment;
- appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);

- equipment cleaning materials;
- appropriate sample containers and labels;
- chain-of-custody forms;
- insulated coolers with ice, when collecting samples requiring preservation by chilling;
- photoionization detector (PID) or flame ionization detector (FID); and
- field notebook and/or personal digital assistant (PDA).

#### IV. Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be identified by one of the following three actions (lines of evidence):

- Contact the State One Call
- Obtain a detailed site utility plan drawn to scale, preferably an "as-built" plan
- Conduct a detailed visual site inspection

In the event that one or more of the above lines of evidence cannot be conducted, or if the accuracy of utility location is questionable, a minimum of one additional line of evidence will be utilized as appropriate or suitable to the conditions. Examples of additional lines of evidence include but are not limited to:

- Private utility locating service
- Research of state, county or municipal utility records and maps including computer drawn maps or geographical information systems (GIS)
- Contact with the utility provider to obtain their utility location records
- Hand augering or digging
- Hydro-knife
- Air-knife

- Radio Frequency Detector (RFD)
- Ground Penetrating Radar (GPR)
- Any other method that may give ample evidence of the presence or location of subgrade utilities.

Overhead power lines also present risks and the following safe clearance must be maintained from them.

Power Line Voltage Phase to Phase (kV)	Minimum Safe Clearance (feet)
50 or below	10
Above 50 to 200	15
Above 200 to 350	20
Above 350 to 500	25
Above 500 to 750	35
Above 750 to 1,000	35

ANSI Standard B30.5-1994, 5-3.4.5

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Specifications of materials used for backfilling borehole will be obtained, reviewed and approved to meet project quality objectives.

#### V. Health and Safety Considerations

Field activities associated with overburden drilling and soil sampling will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

#### VI. Procedure

#### **Drilling Procedures**

The drilling contractor will be responsible for obtaining accurate and representative samples; informing the supervising geologist of changes in drilling pressure; and keeping a separate general log of soils encountered, including blow counts (i.e., the number of blows from a soil sampling drive weight [140 pounds] required to drive the split-barrel sampler in 6-inch increments). Records will also be kept of occurrences of premature refusal due to boulders or construction materials that may have been used as fill. Where a boring cannot be advanced to the desired depth, the boring will be abandoned and an additional boring will be advanced at an adjacent location to obtain the required sample. Where it is desirable to avoid leaving vertical connections between depth intervals, the borehole will be sealed using cement and/or bentonite. Multiple refusals may lead to a decision by the supervising geologist to abandon that sampling location.

#### Soil Sampling Procedures

Samples of subsurface materials encountered while drilling soil borings will be collected using one of the following methods:

- 2-inch split-barrel (split-spoon) sampler, if using the ASTM D 1586 Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils
- Plastic internal soil sample sleeves if using direct-push drilling.

Soil samples are typically field screened with an FID or PID at sites where volatile organic compounds are present in the subsurface. Field screening is performed using one of the following methods:

- Upon opening the sampler, the soil is split open and the PID or FID probe is placed in the opening and covered with a gloved hand. Such readings should be obtained at several locations along the length of the sample
- A portion of the collected sample is placed in a jar, which is covered with aluminum foil, sealed, and allowed to warm to room temperature. After warming, the cover is removed, the foil is pieced with the FID or PID probe, and a reading is obtained.

Samples selected for laboratory analysis will be handled, packed, and shipped in accordance with the procedures outlined in the Work Plan or Chain-of-Custody, Handling, Packing, and Shipping SOP.

A geologist will be onsite during drilling and sampling operations to describe each soil sample on the soil boring log, including:

- percent recovery;
- structure and degree of sample disturbance;
- soil type;
- color;
- moisture condition;
- density;
- grain-size;
- consistency; and
- other observations, particularly relating to the presence of waste materials

Further details regarding geologic description of soil samples are presented in the Soil Description SOP.

Particular care will be taken to fully describe any sheens observed, oil saturation, staining, discoloration, evidence of chemical impacts, or unnatural materials.

#### VII. Waste Management

Water generated during cleaning procedures will be collected and contained onsite in appropriate containers for future analysis and appropriate disposal.

PPE (such as gloves, disposable clothing, and other disposable equipment) resulting from personnel cleaning procedures and soil sampling/handling activities will be placed in plastic bags. These bags will be transferred into appropriately labeled 55-gallon drums or a covered roll-off box for appropriate disposal.

Soil materials will be placed in sealed 55-gallon steel drums or covered roll-off boxes and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

#### VIII. Data Recording and Management

The supervising geologist or scientist will be responsible for documenting drilling events using a bound field notebook and/or PDA to record all relevant information in a clear and concise format. The record of drilling events will include:

- start and finish dates of drilling;
- name and location of project;
- project number, client, and site location;
- sample number and depths;
- blow counts and recovery;
- depth to water;
- type of drilling method;
- drilling equipment specifications, including the diameter of drilling tools;
- documentation of any elevated organic vapor readings;
- names of drillers, inspectors, or other people onsite; and
- weather conditions.

#### IX. Quality Assurance

Equipment will be cleaned prior to use onsite, between each drilling location, and prior to leaving the site. Drilling equipment and associated tools, including augers, drill rods, sampling equipment, wrenches, and other equipment or tools that may have come in contact with soils and/or waste materials will be cleaned with high-pressure steam-cleaning equipment using a potable water source. The drilling equipment will be cleaned in an area designated by the supervising engineer or geologist that is located outside of the work zone. More elaborate cleaning procedures may be

required for reusable soil samplers (split-spoons) when soil samples are obtained for laboratory analysis of chemical constituents.

#### X. References

American Society of Testing and Materials (ASTM) D 1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils.* 



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### Photoionization Detector Air Monitoring and Field Screening

Rev. #: 1

Rev Date: November 8, 2009

SOP: Photoionization Detector Air Monitoring and Field Screening Rev. #: 0 | Rev Date: July 28, 2003

**Approval Signatures** 

Prepared by: (the late) Maureen Geisser

Date: July 28, 2003

Christipto ( the

Reviewed/revised by: Christopher C. Lutes (Technical Expert)

Date: November 8, 2009

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#### I. Scope and Application

Field screening with a photoionization detector (PID), such as an HNu<sup>™</sup>, Photovac<sup>™</sup>, MicroTIP<sup>™</sup>, or MiniRAE<sup>™</sup>, is a procedure to measure relative concentrations of volatile organic compounds (VOCs) and other compounds. Characteristics of the PID are presented in Attachment 1 and the compounds a PID can detect are presented in Attachment 2. Field screening will frequently be conducted on the following:

- Work area air to assess exposure to on-site workers of air contaminants via the air pathway;
- Well headspaces as a precautionary measure each time the well cover is opened; and
- Headspace of soil samples to assess the relative concentration of volatile organics in the sample or to select particular intervals for off-site analysis for VOCs.

#### II. Personnel Qualifications

Personnel performing this method should be familiar with the basic principles of quantiative analytical chemistry (such as calibration) and familiar with the particular operation of the instrument to be used.

#### III. Equipment List

The following materials, as required, shall be available while performing PID field screening:

- personal protective equipment (PPE), as required by the site Health and Safety Plan (HASP);
- PID and operating manual;
- PID extra battery pack and battery charger;
- calibration canisters for the PID;
- sample jars;
- Q-tips;

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- aluminum foil;
- field calibration log (attached); and
- field notebook.

#### IV. Cautions

PIDs are sensitive to moisture and may not function under high humidity. PIDs cannot be used to indicate oxygen deficiency or combustible gases.

#### V. Health and Safety Considerations

Since the PIDs cannot detect all of the chemicals that may be present at a sample location, a zero reading on either instrument does not necessarily signify the absence of air contaminants. PIDs cannot be used as an indicator for oxygen deficiency.

VI. Procedure (Note these procedures were written particular to one specific instrument model, therefore please also refer to your owners manual. Hhowever the general principles – such as always measuring both a zero and span gas after an instrument adjustment/at the beginning of the analytical day, after four hours of testing and again at the end of an analytical day can be applied to all instruments.)

#### **PID** Calibration

PID field instruments will be calibrated and operated to yield "total organic vapor" in parts per million (ppm) (v/v) relative to benzene or isobutylene (or equivalent). Operation, maintenance, and calibration shall be performed in accordance with the manufacturer's instructions and entered on the PID calibration and maintenance log (Attachment 3).

- 1. Don PPE, as required by the HASP.
- 2. Perform a BATTERY CHECK. Turn the FUNCTION switch to the BATTERY CHECK position. Check that the indicator is within or beyond the green battery arc. If battery is low, the battery must be charged before calibration.
- 3. Allow the instrument to warm up, then calibrate the PID. If equipped, turn the FUNCTION switch to the STANDBY position and rotate the ZERO

POTENTIOMETER until the meter reads zero with the instrument sampling clean air. Wait 15 to 20 seconds to confirm the adjustment. If unstable, readjust. If equipped, check to see that the SPAN POTENTIOMETER is adjusted for the probe being used (e.g., 9.8 for 10.2 electron volts [eV]). Set the FUNCTION switch to the desired ppm range (0-20, 0-200, or 0-2,000). A violet glow from the ultraviolet (UV) source should be visible at the sample inlet of the probe/sensor unit.

- 4. Listen for the fan operation to verify fan function.
- 5. Connect one end of the sampling hose to the calibration canister regulator outlet and the other end to the sampling probe of the PID. Crack the regulator valve and take a reading after 5 to 10 seconds. Adjust the span potentiometer to produce the concentration listed on the span gas cylinder. Record appropriate information on a PID Calibration and Maintenance Log (Attachment 3, or equivalent).
- 6. If so equipped, set the alarm at desired level.
- 7. Recheck the zero with fresh/clean air
- 8. Always recheck both zero and span after making any instrment adjustment, after four hours of screenign work and again after sample analysis.

#### Work Area Air Monitoring

- 1. Measure and record the background PID reading.
- 2. Measure and record the breathing space reading.

#### Well Headspace Screening

- 1. Measure and record the background PID reading.
- 2. Unlock and open the well cover while standing upwind of the well.
- 3. Remove the well cap.
- 4. Place the PID probe approximately 6 inches above the top of the casing.
- 5. Record all PID readings and proceed in accordance with the HASP.

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#### **Field Screening Procedures**

Soil samples will be field screened upon collection with the PID for a relative measure of the total volatile organic concentration. The following steps define the PID field screening procedures.

- Half-fill two clean glass jars with the sample (if sufficient quantities of soil are available) to be analyzed. Quickly cover each open top with one or two sheets of clean aluminum foil and subsequently apply screw caps to tightly seal the jars. Sixteen-ounce (approximately 500 mL) soil or "mason" type jars are preferred; jars less than 8 ounces (approximately 250 mL) total capacity may not be used.
- Allow headspace development for at least 10 minutes. Vigorously shake jars for 15 seconds at both the beginning and end of the headspace development period. Where ambient temperatures are below 32°F (0°C), headspace development should be within a heated building.
- Subsequent to headspace development, remove screw lid to expose the foil seal. Quickly puncture foil seal with instrument sampling probe, to a point about one-half of the headspace depth. Exercise care to avoid contact with water droplets or soil particulates.
- 4. Following probe insertion through foil seal, record the highest meter response for each sample as the jar headspace concentration. Using the foil seal/probe insertion method, maximum response should occur between 2 and 5 seconds. Erratic meter response may occur at high organic vapor concentrations or conditions of elevated headspace moisture, in which case headspace data should be recorded and erratic meter response noted.
- 5. The headspace screening data from both jar samples should be recorded and compared; generally, replicate values should be consistent to plus or minus 20%. It should be noted that in some cases (e.g., 6-inch increment soil borings), sufficient sample quantities may not be available to perform duplicate screenings. One screening will be considered sufficient for this case.
- 6. PID field instruments will be operated and calibrated to yield "total organic vapors" in ppm (v/v) as benzene. PID instruments must be operated with at least a 10.0 eV (+) lamp source. Operation, maintenance, and calibration will be performed in accordance with the manufacturer's specifications presented in Attachment 12-1. For jar headspace analysis, instrument calibration will be checked/adjusted at least twice per day, at the beginning and end of each day

of use. Calibration will exceed twice per day if conditions and/or manufacturer's specifications dictate.

 Instrumentation with digital (LED/LCD) displays may not be able to discern maximum headspace response unless equipped with a "maximum hold" feature or strip-chart recorder.

#### VII. Waste Management

Do not dispose canisters of compressed gas, if there is still compressed gas in the canister. Return the canister to the manufactuer for proper disposal.

#### VIII. Data Recording and Management

Measurements will be record in the field notebook or boring logs at the time of measurement with notation of date, time, location, depth (if applicable), and item monitored. If a data memory is available, readings will be downloaded from the unit upon access to a computer with software to retrieve the data.

#### IX. Quality Assurance

After each use, the readout unit should be wiped down with a clean cloth or paper towel.

For a HNu, the UV light source window and ionization chamber should be cleaned once a month in the following manner:

- 1. With the PID off, disconnect the sensor/probe from the unit.
- 2. Remove the exhaust screw, grasp the end cap in one hand and the probe shell in the other, and pull apart.
- 3. Loosen the screws on top of the end cap and separate the end cap and ion chamber from the lamp and lamp housing.
- 4. Tilt the lamp housing with one hand over the opening so that the lamp slides out into your hand.
- 5. Clean the lamp with lens paper and HNu cleaning compound (except 11.7 eV). For the 11.7 eV lamp, use a chlorinated organic solvent.

- 6. Clean the ion chamber using methanol on a Q-tip and then dry gently at 50°C to 60°C for 30 minutes.
- 7. Following cleaning, reassemble by first sliding the lamp back into the lamp housing. Place ion chamber on top of the housing, making sure the contacts are properly aligned.
- 8. Place the end cap on top of the ion chamber and replace the two screws (tighten the screws only enough to seal the o-ring).
- 9. Line up the pins on the base of the lamp housing with pins inside the probe shell and slide the housing assembly into the shell.

#### X. References

Denahan, S.A. et. all "Relationships Between Chemical Screening Methodologies for Petroleum Contaminated Soils: Theory and Pracice" *Chapter 5 In Principles and Practices for Petroleum Contaminated Soils,* E.J. Calabrese and P.T. Kostecki Eds., Lewis Publishers 1993.

Fitzgerald, J. "Onsite Analytical Screening of Gasoline Contaminated Media Using a Jar Headspace Procedure" Chapter 4 *in Principles and Practices for Petroleum Contaminated Soils,* E.J. Calabrese and P.T. Kostecki Eds., Lewis Publishers 1993.

1

#### **ATTACHMENT 1**

Characteristics of the Photoionization Detector (PID)

#### I. Introduction

PIDs are used in the field to detect a variety of compounds in air. PIDs can be used to detect leaks of volatile substances in drums and tanks, to determine the presence of volatile compounds in soil and water, and to make ambient air surveys. If personnel are thoroughly trained to operate the instrument and interpret the data, these PID instruments can be a valuable tool. Its use can help in deciding the level of protection to be worn, assist in determining the implementation of other safety procedures, and in determining subsequent monitoring or sampling locations.

Portable PIDs detect the concentration of organic gases, as well as a few inorganic gases. The basis for detection is the ionization of gaseous species. The incoming gas molecules are subjected to UV radiation, which ionizes molecules that have an ionization potential (IP) less than or equal to that rated for the UV source. Every molecule has a characteristic IP, which is the energy required to remove an electron from the molecule, thus yielding a positively charged ion and the free electron. These ions are attracted to an oppositely charged electrode, causing a current and an electric signal to the LED display. Compounds are measured on a ppm volume basis.

#### II. HNu PI-101 / MiniRAE or Equivalent PID

The PIDs detect the concentration of organic gases, as well as a few inorganic gases. The basis for detection is the ionization of gaseous species. The incoming gas molecules are subjected to UV radiation, which is energetic enough to ionize many gaseous compounds. Each molecule is transformed into charged ion pairs, creating a current between two electrodes. Every molecule has a characteristic IP, which is the energy required to remove an electron from the molecule, yielding a positively charged ion and the free electron.

Three probes, each containing a different UV light source, are available for use with the PID. Probe energies are typically 9.5, 10.2, and 11.7 eV, respectively. All three probes detect many aromatic and large-molecule hydrocarbons. In addition, the 10.2 eV and 11.7 eV probes detect some smaller organic molecules and some halogenated hydrocarbons. The 10.2 eV probe is the most useful for environmental response work, as it is more durable than the 11.7 eV probe and detects more compounds than the 9.5 eV probe. A listing of molecules and compounds that the HNu can detect is presented in Attachment 2.

The primary PID calibration gas is either benzene or isobutylene. The span potentiometer knob is turned to 9.8 for benzene calibration. A knob setting of zero increases the sensitivity to benzene approximately 10-fold. Its lower detection limit is in the low ppm range. Additionally, response time is rapid; the dot matrix liquid crystal displays 90% of the indicated concentration within 3 seconds.

#### III. Limitations

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The PID instrument can monitor several vapors and gases in air. Many non-volatile liquids, toxic solids, particulates, and other toxic gases and vapors, however, cannot be detected with PIDs (such as methane). Since the PIDs cannot detect all of the chemicals that may be present at a sample location, a zero reading on either instrument does not necessarily signify the absence of air contaminants.

The PID instrument is generally not specific and their response to different compounds is relative to the calibration gases. Instrument readings may be higher or lower than the true concentration. This effect can be observed when monitoring total contaminant concentrations if several different compounds are being detected at once. In addition, the response of these instruments is not linear over the entire detection range. Therefore, care must be taken when interpreting the data. Concentrations should be reported in terms of the calibration gas and probe type.

PIDs are small, portable instruments and may not yield results as accurate as laboratory instruments. PIDs were originally designed for specific industrial applications. They are relatively easy to use and interpret when detecting total concentrations of known contaminants in air, but interpretation becomes more difficult when trying to identify the individual components of a mixture. PIDs cannot be used as an indicator for combustible gases or oxygen deficiency.

#### **ATTACHMENT 2**

#### Molecules and Compounds Detected by a PID

Some Atoms and Simple Molecules		<u>mple Molecules</u>	Paraffins and Cycloparaffins	
	<u>IP(eV)</u>	<u>IP(eV)</u>	Molecule	<u>IP(eV)</u>
H C N	13.595 I₂ 11.264 HF 14.54 HCl	9.28 15.77 12.74	methane ethane	12.98 11.65 11.07
O Si	13.614 HBr 8.149 HI	11.62 10.38	propane n-butane i-butane	10.63 10.57
S F	10.357 SO <sub>2</sub> 17.42 CO <sub>2</sub>	12.34 13.79	n-pentane i-pentane	10.35 10.32
CI Br	13.01 COS 11.84 CS <sub>2</sub>	11.18 10.08	2,2-dimethylpropane n-hexane	10.35 10.18
I H <sub>2</sub> N <sub>2</sub>	10.48 N <sub>2</sub> O 15.426 NO <sub>2</sub> 15.580 O <sub>3</sub>	12.90 9.78 12.80	2-methlypentane 3-methlypentane 2,2-dimethlybutane	10.12 10.08 10.06
O <sub>2</sub> CO	12.075 H <sub>2</sub> O 14.01 H <sub>2</sub> S	12.59 10.46	2,3-dimethlybutane n-heptane	10.02 10.08
CN NO CH	15.13 H₂Se 9.25 H₂Te 11.1 HCN	9.88 9.14 2.01	2,2,4-trimethlypentane cyclopropane	9.86 10.06
OH F <sub>2</sub>	13.18 C <sub>2</sub> N <sub>2</sub> 15.7 NH <sub>3</sub>	3.91 13.8 10.15	cyclopentane cyclohexane methlycyclohexane	10.53 9.88 9.8
Cl <sub>2</sub> Br <sub>2</sub>	11.48 CH₃ 10.55 CH₄	9.840 12.98		

#### Alkyl Halides

Alkyl Halides

<u>IP(eV)</u>	<u>IP(eV)</u>	<u>Molecule</u>	<u>IP(eV)</u>
HCI	12.74	methyl iodide	9.54
Cl <sub>2</sub>	11.48	diiodomethane	9.34
CH <sub>4</sub>	12.98	ethyl iodide	9.33
methyl chloride	11.28	1-iodopropane	9.26
dichloroemethane	11.35	2-iodopropane	9.17
trichloromethane	11.42	1-iodobutane	9.21
tetrachloromethane	11.47	2-iodobutane	9.09
ethyl chloride	10.98	1-iodo-2-methylpropane	9.18
1,2-dichloroethane	11.12	2-iodo-2-methylpropane	9.02
1-chloropropane	10.82	1-iodopentane	9.19
2-chloropropane	10.78	F <sub>2</sub>	15.7
1,2-dichloropropane	10.87	HF	15.77
1,3-dichloropropane	10.85	CFCI <sub>3</sub> (Freon 11)	11.77
1-chlorobutane	10.67	CF <sub>2</sub> Cl <sub>2</sub> (Freon 12)	12.31
2-chlorobutane	10.65	CF <sub>3</sub> CI (Freon 13)	12.91
1-chloro-2-methylpropane	10.66	CHCIF <sub>2</sub> (Freon 22)	12.45
2-chloro-2-methylpropane	10.61	CFBR <sub>3</sub>	10.67
HBr	11.62	$CF_2Br_2$	11.07
Br <sub>2</sub>	10.55	CH <sub>3</sub> CF <sub>2</sub> CI (Genetron 101)	11.98
methyl bromide	10.53	CFCI <sub>2</sub> CF <sub>2</sub> CI	11.99
dibromomethane	10.49	CF <sub>3</sub> CCl <sub>3</sub> (Freon 113)	11.78
tribromomethane	10.51	CFHBrCH <sub>2</sub> Cr	10.75
CH₂BrCl	10.77	$CF_2BrCH_2Br$	10.83
CHBr <sub>2</sub> Cl	10.59	CF <sub>3</sub> CH <sub>2</sub> I	10.00
ethyl bromide	10.29	n-C <sub>3</sub> F <sub>7</sub> I	10.36
1,1-dibromoethane	10.19	n-C <sub>3</sub> F <sub>7</sub> CH <sub>2</sub> Cl	11.84
1-bromo-2-chloroethane	10.63	n-C <sub>3</sub> F <sub>7</sub> CH <sub>2</sub> I	9.96
1-bromopropane	10.18		
2-bromopropane	10.075		
1,3-dibromopropane	10.07		
1-bromobutane	10.13		
2-bromobutane	9.98		
1-bromo-2-methylpropane	10.09		
2-bromo-2-methylpropane	9.89		
1-bromopentane	10.10		
HI	10.38		
l <sub>2</sub>	9.28		

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#### Aliphatic Alcohol, Ether, Thiol, and Sulfides

Molecule	<u>IP(eV)</u>
H <sub>2</sub> O	12.59
methyl alcohol	10.85
ethyl alcohol	10.48
n-propyl alcohol	10.20
i-propyl alcohol	10.16
n-butyl alcohol	10.04
dimethyl ether	10.00
diethyl ether	9.53
n-propyl ether	9.27
i-propyl ether	9.20
H <sub>2</sub> S	10.46
methanethiol	9.440
ethanethiol	9.285
1-propanethiol	9.195
1-butanethiol	9.14
dimethyl sulfide	8.685
ethyl methyl sulfide	8.55
diethyl sulfide	8.430
di-n-propyl sulfide	8.30

#### Aliphatic Aldehydes and Ketones

#### Aliphatic Acids and Esters

<u>Molecule</u>	IP(eV)	Molecule	<u>IP(eV)</u>
CO <sub>2</sub>	13.79	CO <sub>2</sub>	13.79
formaldehyde	10.87	formic acid	11.05
acetaldehyde	10.21	acetic acid	10.37
propionaldehyde	9.98	propionic acid	10.24
n-butyraldehyde	9.86	n-butyric acid	10.16
isobutyraldehyde	9.74	isobutyric acid	10.02
n-valeraldehyde	9.82	n-valeric acid	10.12
isovaleraldehyde	9.71	methyl formate	10.815
acrolein	10.10	ethyl formate	10.61
crotonaldehyde	9.73	n-propyl formate	10.54
benzaldehyde	9.53	n-butyl formate	10.50
acetone	9.69	isobutyl formate	10.46
methyl ethyl ketone	9.53	methyl acetate	10.27
methyl n-propyl ketone	9.39	ethyl acetate	10.11
methyl i-propyl ketone	9.32	n-propyl acetate	10.04
diethyl ketone	9.32	isopropyl acetate	9.99
methyl n-butyl ketone	9.34	n-butyl acetate	10.01
methyl i-butyl ketone	9.30	isobutyl acetate	9.97
3,3-dimethyl butanone	9.17	sec-butyl acetate	9.91
2-heptanone	9.33	methyl propionate	10.15
cyclopentanone	9.26	ethyl propionate	10.00
cyclohexanone	9.14	methyl n-butyrate	10.07
2,3-butanedione	9.23	methyl isobutyrate	9.98
2,4-pentanedione	8.87		

### Aliphatic Amines and Amides

### Other Aliphatic Molecules with N Atom

<u>Molecule</u>	<u>IP(eV)</u>
NH <sub>3</sub>	10.15
methyl amine	8.97
ethyl amine	8.86
n-propyl amine	8.78
i-propyl amine	8.72
n-butyl amine	8.71
i-butyl amine	8.70
s-butyl amine	8.70
t-butyl amine	8.64
dimethyl amine	8.24
diethyl amine	8.01
di-n-propyl amine	7.84
di-i-propyl amine	7.73
di-n-butyl amine	7.69
trimethyl amine	7.82
triethyl amine	7.50
tri-n-propyl amine	7.23
formamide	10.25
acetamide	9.77
N-methyl acetamide	8.90
N,N-dimethyl formamide	9.12
N,N-dimethyl acetamide	8.81
N,N-diethyl formamide	8.89
N,N-diethyl acetamide	8.60

<u>Molecule</u>	<u>IP(eV)</u>
nitromethane	11.08
nitroethane	10.88
1-nitropropane	10.81
2-nitropropane	10.71
HCN	13.91
acetonitrile 12.22	
propionitrile	11.84
n-butyronitrile	11.67
acrylonitrile	10.91
3-butene-nitrile	10.39
ethyl nitrate	11.22
n-propyl nitrate	
methyl thiocyanate	10.065
ethyl thiocyanate	9.89
methyl isothiocyanate	9.25
ethyl isothiocyanate	9.14

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### Olefins, Cyclo-ofefins, Acetylenes

### Some Derivatives of Olefins

<u>Molecule</u>	<u>IP(eV)</u>	<u>Molecule</u>	<u>IP(eV)</u>
ethylene	10.515	vinyl chloride	9.995
propylene	9.73	cis-dichloroethylene	9.65
1-butene	9.58	trans-dichloroethylene	9.66
2-methylpropene	9.23	trichloroethylene	9.45
trans-2-butene	9.13	tetrachloroethylene	9.32
cis-2-butene	9.13	vinyl bromide	9.80
1-pentene	9.50	1,2-dibromoethylene	9.45
2-methyl-1-butene	9.12	tribromoethylene	9.27
3-methyl-1-butene	9.51	3-chloropropene	10.04
3-methyl-2-butene	8.67	2,3-dichloropropene	9.82
1-hexene	9.46	1-bromopropene	9.30
1,3-butadiene	9.07	3-bromopropene	9.7
isoprene	8.845	CF <sub>3</sub> CCI=CCICF <sub>3</sub>	10.36
cyclopentene	9.01	$n-C_5F_{11}CF=CF_2$	10.48
cyclohexene	8.945	acrolein	10.10
4-methylcyclohexene	8.91	crotonaldehyde	9.73
4-cinylcylohexene	8.93	mesityl oxide	9.08
cyclo-octatetraene	7.99	vinyl methyl ether	8.93
acetylene	11.41	allyl alcohol	9.67
propyne	10.36	vinyl acetate	9.19
1-butyne	10.18		

### Aromatic Compounds

### Aromatic Compounds

<u>Molecule</u>	<u>IP(eV)</u>	Molecule	<u>IP(eV)</u>
benzene	9.245	phenyl isothiocyanate	8.520
toluene	8.82	benzonitrile	9.705
ethyl benzene	8.76	nitrobenzene	9.92
n-propyl benzene	8.72	aniline	7.70
i-propyl benzene	8.69	fluoro-benzene	9.195
n-butyl benzene	8.69	chloro-benzene	9.07
s-butyl benzene	8.68	bromo-benzene	8.98
t-butyl benzene	8.68	iodo-benzene	8.73
o-xylene	8.56	o-dichlorobenzene	9.07
m-xylene	8.56	m-dichlorobenzene	9.12
p-xylene	8.445	p-dichlorobenzene	8.94
mesitylene	8.40	1-chloro-2-fluorobenzene	9.155
durene	8.025	1-chloro-3-fluorobenzene	9.21
styrene	8.47	1-chloro-4-fluorobenzene	8.99
alpha-methyl styrene	8.35	o-fluorotoluene	8.915
ethynylbenzene	8.815	m-fluorotoluene	8.915
naphthalene	8.12	p-fluorotoluene	8.785
1-methylnapthalene	7.69	o-chlorotoluene	8.83
2-methylnapthalene	7.955	m-chlorotoluene	8.83
biphenyl	8.27	p-chlorotoluene	8.70
phenol	8.50	o-bromotoluene	8.79
anisole	8.22	m-bromotoluene	8.81
phenetole	8.13	p-bromotoluene	8.67
benzaldehyde	9.53	o-iodotoluene	8.62
acetophenone	9.27	m-iodotoluene	8.61
benzenethiol	8.33	p-iodotoluene	8.50
phenyl isocyanate	8.77	benzotrifluoride	9.68
		o-fluorophenol	8.66

### Heterocyclic Molecules

#### **Miscellaneous Molecules**

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Molecule	<u>IP(eV)</u>	Molecule	<u>IP(eV)</u>
furan	8.89	ethylene oxide	10.565
2-methyl furan	8.39	propylene oxide	10.22
2-furaldehyde	9.21	p-dioxane	9.13
tetrahydrofuran	9.54	dimethoxymethane	10.00
dihydropyran	8.34	diethoxymethane	9.70
tetrahydropyran	9.26	1,1-dimethoxyethane	9.65
thiophene	8.860	propiolactone	9.70
2-chlorothiophene	8.68	methyl disulfide	8.46
2-bromothiophene	8.63	ethyl disulfide	8.27
pyrrole	8.20	diethyl sulfite	9.68
pyridine	9.32	thiolacetic acid	10.00
2-picoline	9.02	acetyl chloride	11.02
3-picoline	9.04	acetyl bromide	10.55
4-picoline	9.04	cyclo-C <sub>6</sub> H <sub>11</sub> CF <sub>3</sub>	10.46
2,3-lutidine	8.85	(n-C <sub>3</sub> F <sub>7</sub> )(CH <sub>3</sub> )C=O	10.58
2,4-lutidine	8.85	trichlorovinylsilane	10.79
2,6-lutidine	8.85	$(C_2F_5)_3N$	11.7
		isoprene	9.08
		phosgene	11.77

#### Notes:

Reference: HNu Systems, Inc., 1985 IP = Ionization Potential SOP: Photoionization Detector Air Monitoring and Field Screening Rev. #: 0 | Rev Date: July 28, 2003

#### **ATTACHMENT 3**

PID CALIBRATION AND MAINTENANCE LOG							
Instrument Mo	odel Number						
Instrument Se	rial Number						
Calibration Ga	as		ppm				
				Calibra	tion		
Date/Time	Initials	Battery Check	Background Value	True Gas Value	Measured Gas Value	Adjust	
COMMENTS:							



Imagine the result

# **Monitoring Well Installation**

Rev. #: 2

Rev Date: August 22, 2008

**Approval Signatures** 

Prepared by: Michael J. Shefull D

Date: 07/22/2010

Date: 8/25/08

(Technical Expert)

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#### I. Scope and Application

The procedures set out herein are designed to produce standard groundwater monitoring wells suitable for: (1) groundwater sampling, (2) water level measurement, (3) bulk hydraulic conductivity testing of formations adjacent to the open interval of the well.

Monitoring well boreholes in unconsolidated (overburden) materials are typically drilled using the hollow-stem auger drilling method. Other drilling methods that are also suitable for installing overburden monitoring wells, and are sometimes necessary due to site-specific geologic conditions, include: drive-and-wash, spun casing, Rotasonic, dual-rotary (Barber Rig), and fluid/mud rotary with core barrel or roller bit. Direct-push techniques (e.g., Geoprobe or cone penetrometer) and driven well points may also be used in some cases within the overburden. Monitoring wells within consolidated materials such as bedrock are commonly drilled using water-rotary (coring or tri-cone roller bit), air rotary or Rotasonic methods. The drilling method to be used at a given site will be selected based on site-specific consideration of anticipated drilling/well depths, site or regional geologic knowledge, type of monitoring to be conducted using the installed well, and cost.

No oils or grease will be used on equipment introduced into the boring (e.g., drill rod, casing, or sampling tools). No polyvinyl chloride (PVC) glue/cement will be used in constructing or retrofitting monitoring wells that will be used for water-quality monitoring. No coated bentonite pellets will be used in the well drilling or construction process. Specifications of materials to be installed in the well will be obtained prior to mobilizing onsite, including:

- well casing;
- bentonite;
- sand; and
- grout.

Well materials will be inspected and, if needed, cleaned prior to installation.

#### II. Personnel Qualifications

Monitoring well installation activities will be performed by persons who have been trained in proper well installation procedures under the guidance of an experienced field geologist, engineer, or technician. Where field sampling is performed for soil or

bedrock characterization, field personnel will have undergone in-field training in soil or bedrock description methods, as described in the appropriate SOP(s) for those activities.

#### III. Equipment List

The following materials will be available during soil boring and monitoring well installation activities, as required:

- Site Plan with proposed soil boring/well locations;
- Work Plan or Field Sampling Plan (FSP), and site Health and Safety Plan (HASP);
- personal protective equipment (PPE), as required by the HASP;
- traffic cones, delineators, caution tape, and/or fencing as appropriate for securing the work area, if such are not provided by drillers;
- appropriate soil sampling equipment (e.g., stainless steel spatulas, knife);
- soil and/or bedrock logging equipment as specified in the appropriate SOPs;
- appropriate sample containers and labels;
- drum labels as required for investigation derived waste handling;
- chain-of-custody forms;
- insulated coolers with ice, when collecting samples requiring preservation by chilling;
- photoionization detector (PID) or flame ionization detector (FID);
- ziplock style bags;
- water level or oil/water interface meter;
- locks and keys for securing the well after installation;
- decontamination equipment (bucket, distilled or deionized water, cleansers appropriate for removing expected chemicals of concern, paper towels);

• field notebook.

Prior to mobilizing to the site, ARCADIS personnel will contact the drilling subcontractor or in-house driller (as appropriate) to confirm that appropriate sampling and well installation equipment will be provided. Specifications of the sampling and well installation equipment are expected to vary by project, and so communication with the driller will be necessary to ensure that the materials provided will meet the project objectives. Equipment typically provided by the driller could include:

- drilling equipment required by the American Society of Testing and Materials (ASTM) D 1586, when performing split-spoon sampling;
- disposable plastic liners, when drilling with direct-push equipment;
- drums for investigation derived waste;
- drilling and sampling equipment decontamination materials;
- decontamination pad materials, if required; and
- well construction materials.

#### IV. Cautions

Prior to beginning field work, underground utilities in the vicinity of the drilling areas will be delineated by the drilling contractor or an independent underground utility locator service. See separate SOP for utility clearance.

Some regulatory agencies require a minimum annular space between the well or permanent casing and the borehole wall. When specified, the minimum clearance is typically 2 inches on all sides (e.g., a 2-inch diameter well requires a 6-inch diameter borehole). In addition, some regulatory agencies have specific requirements regarding grout mixtures. Determine whether the oversight agency has any such requirements prior to finalizing the drilling and well installation plan.

If dense non-aqueous phase liquids (DNAPL) are known or expected to exist at the site, refer to the DNAPL Contingency Plan SOP for additional details regarding drilling and well installation to reduce the potential for inadvertent DNAPL remobilization.

Avoid using drilling fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

Similarly, consider the material compatibility between the well materials and the surrounding environment. For example, PVC well materials are not preferred when DNAPL is present. In addition, some groundwater conditions leach metals from stainless steel.

Water used for drilling and sampling of soil or bedrock, decontamination of drilling/sampling equipment, or grouting boreholes upon completion will be of a quality acceptable for project objectives. Testing of water supply should be considered.

Specifications of materials used for backfilling bore hole will be obtained, reviewed and approved to meet project quality objectives. Bentonite is not recommended where DNAPLs are likely to be present. In these situations, neat cement grout is preferred.

No coated bentonite pellets will be used in monitoring well construction, as the coating could impact the water quality in the completed well.

Monitoring wells may be installed with Schedule 40 polyvinyl chloride (PVC) to a maximum depth of 200 feet below ground surface (bgs). PVC monitoring wells between 200 and 400 feet total depth will be constructed using Schedule 80 PVC. Monitoring wells deeper than 400 feet will be constructed using steel.

#### V. Health and Safety Considerations

Field activities associated with monitoring well installation will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

#### VI. Procedures

The procedures for installing groundwater monitoring wells are presented below:

Hollow-Stem Auger, Drive-and-Wash, Spun Casing, Fluid/Mud Rotary, Rotasonic, and Dual-Rotary Drilling Methods

- 1. Locate boring/well location, establish work zone, and set up sampling equipment decontamination area.
- Advance boring to desired depth. Collect soil and/or bedrock samples at appropriate interval as specified in the Work Plan and/or FSP. Collect, document, and store samples for laboratory analysis as specified in the Work Plan and/or FSP. Decontaminate equipment between samples in accordance with the Work Plan and/or FSP. A common sampling method that produces

high-quality soil samples with relatively little soil disturbance is the ASTM D 1586 - Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils. Split-spoon samples are obtained during drilling using hollow-stem auger, drive-and-wash, spun casing, and fluid/mud rotary. Rotasonic drilling produces large-diameter soil cores that tend to be more disturbed than split-spoon samples due to the vibratory action of the drill casing. Dual-rotary removes cuttings by compressed air and allows only a general assessment of geology. High-quality bedrock samples can be obtained by coring.

- 3. Describe each soil or bedrock sample as outlined in the appropriate SOP. Record descriptions in the field notebook and/or personal digital assistant (PDA). It should be noted that PDA logs must be electronically backed up and transferred to a location accessible to other project team members as soon as feasible to retain and protect the field data. During soil boring advancement, document all drilling events in field notebook, including blow counts (number of blows required to advance split-spoon sampler in 6-inch increments) and work stoppages. Blow counts will not be available if Rotasonic, dual-rotary, or directpush methods are used. When drilling in bedrock, the rate of penetration (minutes per foot) is recorded.
- 4. If it is necessary to install a monitor well into a permeable zone below a confining layer, particularly if the deeper zone is believed to have water quality that differs significantly from the zone above the confining layer, then a telescopic well construction should be considered. In this case, the borehole is advanced approximately 3 to 5 feet into the top of the confining layer, and a permanent casing (typically PVC, black steel or stainless steel) is installed into the socket drilled into the top of the confining layer. The casing is then grouted in place. The preferred methods of grouting telescoping casings include: pressure-injection grouting using an inflatable packer installed temporarily into the base of the casing, such that grout is injected out the bottom of the casing until it is observed at ground surface outside the casing; displacement-method grouting (also known as the Halliburton method), which entails filling the casing with grout and displacing the grout out the bottom of the casing by pushing a drillable plug, typically made of wood to the bottom of the casing, following by tremie grouting the remainder of the annulus outside the casing; or tremie grouting the annulus surrounding the casing using a tremie pipe installed to the base of the borehole. In all three cases, the casing is grouted to the ground surface, and the grout is allowed to set prior to drilling deeper through the casing. Site-specific criteria and work plans should be created for the completion of non-standard monitoring wells, including telescopic wells.

- 5. In consolidated formations such as competent bedrock, a monitoring well may be completed with an open borehole interval without a screen and sandpack. In these cases, the borehole is advanced to the targeted depth of the top of the open interval. A permanent casing is then grouted in place following the procedures described in Step 4 above. After the grout sets, the borehole is advanced by drilling through the permanent casing to the targeted bottom depth of the open interval, which then serves as the monitoring interval for the well. If open-borehole interval stability is found to be questionable or if a specific depth interval is later selected for monitoring, a screened monitoring well may later be installed within the open-borehole interval, depending on the annular space and well diameter requirements.
- 6. Prior to screened well installation or after the completion of an open-bedrock well, the water level or oil/water interface probe should be used to determine the static water level in the borehole in relation to the proposed well screen or openinterval location. If necessary, an open-bedrock well may be drilled deeper to intersect the water table or a permeable water-bearing zone.
- 7. Upon completing the borehole to the desired depth, if a screened well construction is desired, install the monitoring well by lowering the screen and casing assembly with sump through the augers or casing. Monitoring wells typically will be constructed of 2-inch-diameter, flush-threaded PVC or stainless steel slotted well screen and blank riser casing. Smaller diameters may be used if wells are installed using direct-push methodology or if multiple wells are to be installed in a single borehole. The screen length will be specified in the Work Plan or FSP based on regulatory requirements and specific monitoring objectives. Monitoring well screens are usually 5 to 10 feet long, but may be up to 25 feet long in very low permeability, thick geologic formations. The screen length will depend on the purpose for the well and the objectives of the groundwater investigation. Typically, the slot size will be 0.010 inch and the sand pack will be 20-40, Morie No. 0, or equivalent. In very fine-grained formations where sample turbidity needs to be minimized, it may be preferred to use a 0.006-inch slot size and 30-65, Morie No. 00, or equivalent sand pack. Alternatively, where monitoring wells are installed in coarse-grained deposits and higher well yield is required, a 0.020-inch slot size and 10-20, Morie No. 1, or equivalent sand pack may be preferred. To the extent practicable, the slot size and sand pack gradation may be predetermined in the Work Plan or FSP based on site-specific grain-size analysis or other geologic considerations or monitoring objectives. A blank sump may be attached below the well screen if the well is being installed for DNAPL recovery/monitoring purposes. If so, the annular space around the sump will be backfilled with neat cement grout to the bottom of the well screen prior to placing the sand pack around the screen. A

blank riser will extend from the top of the screen to approximately 2.5 feet above grade or, if necessary, just below grade where conditions warrant a flush-mounted monitoring well. For wells greater than 50 feet deep, centralizers may be desired to assist in centralizing the monitoring well in the borehole during construction.

- 8. When the monitoring well assembly has been set in place and the grout has been placed around the sump (if any), place a washed silica sand pack in the annular space from the bottom of the boring to a height of 1 to 2 feet above the top of the well screen. The sand pack is placed and drilling equipment extracted in increments until the top of the sand pack is at the appropriate depth. The sand pack will be consistent with the screen slot size and the soil particle size in the screened interval, as specified in the Work Plan or FSP. A hydrated bentonite seal (a minimum of 2 feet thick) will then be placed in the annular space above the sand pack. If non-hydrated bentonite is used, the bentonite should be permitted to hydrate in place for a minimum of 30 minutes before proceeding. No coated bentonite pellets will be used in monitoring well drilling or construction. Potable water may be added to hydrate the bentonite if the seal is above the water table. Monitor the placement of the sand pack and bentonite with a weighted tape measure. During the extraction of the augers or casing, a cement/bentonite or neat cement grout will be placed in the annular space from the bentonite seal to a depth approximately 2 feet bgs.
- 9. Place a locking, steel protective casing (extended at least 1.5 feet below grade and 2 feet above grade) over the riser casing and secure with a neat cement seal. Alternatively, for flush-mount completions, place a steel curb box with a bolt-down lid over the riser casing and secure with a neat cement seal. In either case, the cement seal will extend approximately 1.5 to 2.0 feet below grade and laterally at least 1 foot in all directions from the protective casing, and should slope gently away to promote drainage away from the well. Monitoring wells will be labeled with the appropriate designation on both the inner and outer well casings or inside of the curb box lid.

When an above-grade completion is used, the PVC riser will be sealed using an expandable locking plug and the top of the well will be vented by drilling a smalldiameter (1/8 inch) hole near the top of the well casing or through the locking plug, or by cutting a vertical slot in the top of the well casing. When a flushmount installation is used, the PVC riser will be sealed using an unvented, expandable locking plug.

- 10. During well installation, record construction details and actual measurements relayed by the drilling contractor and tabulate materials used (e.g., screen and riser footages; bags of bentonite, cement, and sand) in the field notebook.
- 11. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section VII below.

#### **Direct-Push Method**

The direct-push drilling method may also be used to complete soil borings and install monitoring wells. Examples of this technique include the Diedrich ESP vibratory probe system, GeoProbe®, or AMS Power Probe® dual-tube system. Environmental probe systems typically use a hydraulically operated percussion hammer. Depending on the equipment used, the hammer delivers 140- to 350-foot pounds of energy with each blow. The hammer provides the force needed to penetrate very stiff/medium dense soil formations. The hammer simultaneously advances an outer steel casing that contains a dual-tube liner for sampling soil. The outside diameter (OD) of the outer casing ranges from 1.75 to 2.4 inches and the OD of the inner sampling tube ranges from 1.1 to 1.8 inches. The outer casing isolates shallow layers and permits the unit to continue to probe at depth. The double-rod system provides a borehole that may be tremie-grouted from the bottom up. Alternatively, the inside diameter (ID) of the steel casing provides clearance for the installation of small-diameter (e.g., 0.75- to 1-inch ID) micro-wells. The procedures for installing monitoring wells in soil using the direct-push method are described below.

- 1. Locate boring/well location, establish work zone, and set up sample equipment decontamination area.
- 2. Advance soil boring to designated depth, collecting samples at intervals specified in the Work Plan. Samples will be collected using dedicated, disposable, plastic liners. Describe samples in accordance with the procedures outlined in Step 3 above. Collect samples for laboratory analysis as specified in the Work Plan and/or FSP.
- 3. Upon advancing the borehole to the desired depth, install the micro-well through the inner drill casing. The micro-well will consist of approximately 1-inch ID PVC or stainless steel slotted screen and blank riser. The sand pack, bentonite seal, and cement/bentonite grout will be installed as described, where applicable, in Step 7 and 8 above.

- 4. Install protective steel casing or flush-mount, as appropriate, as described in Step 9 above. During well installation, record construction details and tabulate materials used.
- 5. After completing the well installation, lock the well, clean the area, and dispose of materials in accordance with the procedures outlined in Section VII below.

#### **Driven Well Point Installation**

Well points will be installed by pushing or driving using a drilling rig or direct-push rig, or hand-driven where possible. The well point construction materials will consist of a 1- to 2-inch-diameter threaded steel casing with either 0.010- or 0.020-inch slotted stainless steel screen. The screen length will vary depending on the hydrogeologic conditions of the site. The casings will be joined together with threaded couplings and the terminal end will consist of a steel well point. Because they are driven or pushed to the desired depth, well points do not have annular backfill materials such as sand pack or grout.

#### VII. Waste Management

Investigation-derived wastes (IDW), including soil cuttings and excess drilling fluids (if used), decontamination liquids, and disposable materials (well material packages, PPE, etc.), will be placed in clearly labeled, appropriate containers, or managed as otherwise specified in the Work Plan, FSP, and/or IDW management SOP.

#### VIII. Data Recording and Management

Drilling activities will be documented in a field notebook. Pertinent information will include personnel present on site, times of arrival and departure, significant weather conditions, timing of well installation activities, soil descriptions, well construction specifications (screen and riser material and diameter, sump length, screen length and slot size, riser length, sand pack type), and quantities of materials used. In addition, the locations of newly-installed wells will be documented photographically or in a site sketch. If appropriate, a measuring wheel or engineer's tape will be used to determine approximate distances between important site features.

The well or piezometer location, ground surface elevation, and inner and outer casing elevations will be surveyed using the method specified in the site Work Plan. Generally, a local baseline control will be set up. This local baseline control can then be tied into the appropriate vertical and horizontal datum, such as the National Geodetic Vertical Datum of 1929 or 1988 and the State Plane Coordinate System. At a minimum, the elevation of the top of the inner casing used for water-level

measurements should be measured to the nearest 0.01 foot. Elevations will be established in relation to the National Geodetic Vertical Datum of 1929. A permanent mark will be placed on top of the inner casing to mark the point for water-level measurements.

#### IX. Quality Assurance

All drilling equipment and associated tools (including augers, drill rods, sampling equipment, wrenches, and any other equipment or tools) that may have come in contact with soil will be cleaned in accordance with the procedures outlined in the appropriate SOP. Well materials will also be cleaned prior to well installation.

#### X. References

American Society of Testing and Materials (ASTM) D 1586 - *Standard Test Method for Penetration Test and Split-Barrel Sampling of Soils.* 



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# **Monitoring Well Development**

Rev. #: 2.2

Rev. Date: March 22, 2010

**Approval Signatures** 

Prepared by:

Date: 03/22/2010

Duil S. Lipon Midel J. Sefll Reviewed by:

Date: 03/22/2010

(Technical Expert)

#### I. Scope and Application

Monitoring wells (or piezometers, well points, or micro-wells) will be developed to clear them of fine-grained sediment to enhance the hydraulic connection between the well and the surrounding geologic formation. Development will be accomplished by evacuating well water by either pumping or bailing. Prior to pumping or bailing, the screened interval will be gently surged using a surge block, bailer, or inertia pump with optional surgeblock fitting as appropriate. Accumulated sediment in the bottom of the well (if present) will be removed by bailing with a bottom-loading bailer or via pumping using a submersible or inertia pump with optional surgeblock fitting. Wells will also be gently brushed with a weighted brush to assist in removing loose debris, silt or flock attached to the inside of the well riser and/or screen prior to development. Pumping methods will be selected based on site-specific geologic conditions, anticipated well yield, water table depth, and groundwater monitoring objectives, and may include one or more of the following:

- submersible pump
- inertial pump (Waterra<sup>™</sup> pump or equivalent)
- bladder pump
- peristaltic pump
- centrifugal pump

When developing a well using the pumping method, the pump (or, with inertial pumps, the tubing) is lowered to the screened portion of the well. During purging, the pump or tubing is moved up and down the screened interval until the well yields relatively clear water.

Submersible pumps have a motor-driven impeller that pushes the groundwater through discharge tubing to the ground surface. Inertial pumps have a check valve at the bottom of stiff tubing which, when operated up and down, lifts water to the ground surface. Bladder pumps have a bottom check valve and a flexible internal bladder that fills from below and is then compressed using pressurized air to force water out the top of the bladder through the discharge tubing to the ground surface. These three types of pumps have a wide range of applicability in terms of well depth and water depth.

Centrifugal and peristaltic pumps use atmospheric pressure to lift water from the well, and therefore can only be practically used where the depth to water is less than 25 feet.

#### II. Personnel Qualifications

Monitoring well development activities will be performed by persons who have been trained in proper well development procedures under the guidance of an experienced field geologist, engineer, or technician.

#### III. Equipment List

Materials for monitoring well development using a pump include the following:

- health and safety equipment, as required by the site Health and Safety Plan (HASP):
- cleaning equipment
- photoionization detector (PID) to measure headspace vapors
- pump
- polyethylene pump discharge tubing
- plastic sheeting
- power source (generator or battery)
- field notebook and/or personal digital assistant (PDA)
- graduated pails
- appropriate containers

- monitoring well keys
- water level indicator

Materials for monitoring well development using a bailer include the following:

- personal protective equipment (PPE) as required by the HASP
- cleaning equipment
- PID to measure headspace vapors
- bottom-loading bailer, sand bailer
- polypropylene or nylon rope
- plastic sheeting
- graduated pails
- appropriate containers
- keys to wells
- field notebook and/or PDA
- water level indicator
- weighted brush for well brushing

#### **IV.** Cautions

Where surging is performed to assist in removing fine-grained material from the sand pack, surging must be performed in a gentle manner. Excessive suction could promote fine-grained sediment entry into the outside of the sand pack from the formation.

Avoid using development fluids or materials that could impact groundwater or soil quality, or could be incompatible with the subsurface conditions.

In some cases it may be necessary to add potable water to a well to allow surging and development, especially for new monitoring wells installed in low permeability formations. Before adding potable water to a well, the Project Manager (PM) must be notified and the PM shall make the decision regarding the appropriateness and applicability of adding potable water to a well during well development procedures. If potable water is to be added to a well as part of development, the potable water source should be sampled and analyzed for constituents of concern, and the results evaluated by the PM prior to adding the potable water to the well. If potable water is added to a well for development purposes, at the end of development the well will be purged dry to remove the potable water, or if the well no longer goes dry then the well will be purged to remove at least three times the volume of potable water that was added.

#### V. Health and Safety Considerations

Field activities associated with monitoring well development will be performed in accordance with a site-specific HASP, a copy of which will be present on site during such activities.

#### VI. Procedure

The procedures for monitoring well development are described below. (Note: Steps 7, 8, and 10 can be performed at the same time using an inertial pump with a surge-block fitting.)

- 1. Don appropriate PPE (as required by the HASP).
- 2. Place plastic sheeting around the well.
- Clean all equipment entering each monitoring well, except for new, disposable materials that have not been previously used.

- 4. Open the well cover while standing upwind of the well, remove well cap. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field notebook. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the PID reading in the breathing zone is below 5 PID units, proceed. If the PID reading is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don the appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings.
- 5. Obtain an initial measurement of the depth to water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.
- 6. Prior to redeveloping older wells that may contain solid particulate debris along the inside of the well casing and screen, gently lower and raise a weighted brush along the entire length of the well screen and riser to free and assist in removing loose debris, silt or flock. Perform a minimum of 4 "passes" along the screened and cased intervals of the well below the static water level in the well. Allow the resulting suspended material to settle for a minimum of one day prior to continuing with redevelopment activities.
- 7. Lower a surge block or bailer into the screened portion of the well. Gently raise and lower the surge block or bailer within the screened interval of the well to force water in and out of the screen slots and sand pack. Continue surging for 15 to 30 minutes.
- 8. Lower a bottom-loading bailer, submersible pump, or inertia pump tubing with check valve to the bottom of the well and gently bounce the bailer, pump, pump tubing on the bottom of the well to collect/remove accumulated sediment, if any. Remove and empty the bailer, if used. Repeat until the bailed/pumped water is free of excessive sediment and the bottom of the well feels solid. Alternatively, measurement of the well depth with a water level indicator can be used to verify that sediment and/or silt has been removed to the extent practicable, based on a comparison with the well installation log or previous measurement of total well depth.
- 9. After surging the well and removing excess accumulated sediment from the bottom of the well, re-measure the depth-to-water and the total well depth from the reference point at the top of the well casing. Record these measurements in the field log book.
- Remove formation water by pumping or bailing. Where pumping is used, measure and record the pre-pumping water level. Operate the pump at a relatively constant rate. Measure the pumping rate using a calibrated container and stop watch, and record the pumping rate in the field log book. Measure and record the water level in the well at least

once every 5 minutes during pumping. Note any relevant observations in terms of water color, visual level of turbidity, sheen, odors, etc. Pump or bail until termination criteria specified in the Site Characterization (SC) Work Plan are reached. Record the total volume of water purged from the well.

- 11. If the well goes dry, stop pumping or bailing and allow well to recover. Resume pumping or bailing when sufficient water has recharged the well.
- 12. Contain all water in appropriate containers.
- 13. When complete, secure the lid back on the well.
- 14. Place disposable materials in plastic bags for appropriate disposal and decontaminate reusable, downhole pump components and/or bailer.

#### VII. Waste Management

Materials generated during monitoring well installation and development will be placed in appropriate labeled containers and disposed of as described in the Work Plan or Field Sampling Plan.

#### VIII. Data Recording and Management

Well development activities will be documented in a proper field notebook and/or PDA. Pertinent information will include personnel present on site; times of arrival and departure; significant weather conditions; timing of well development activities; development method(s); observations of purge water color, turbidity, odor, sheen, etc.; purge rate; and water levels before and during pumping.

#### IX. Quality Assurance

All reused, non-disposable, downhole well development equipment will be cleaned in accordance with the procedures outlined in the Field Equipment Cleaning-Decontamination SOP.

### X. References

Not applicable.



#### MONITORING WELL DEVELOPMENT LOG

Sampling Personnel:					Well ID.				
Job Number:					Date:				
Weather:					Time In:		Time Ou	it:	
WELL INFORMATION		TIC	TOC	BGS	check whe	re appropriate :: Flu	ushmount	Stick-Up	
Well Depth	(feet)				Well Lock	ed:	Yes 🔲	No	
Water Table Depth	(feet)				Measurin	g Point Marked:	Yes	No	
					Well Dian	neter:	1"	2"	Other:
WELL WATER INFORMATION									
Length of Water Column:	(feet)				Conversion Factor	3			
Volume of Water in Well:	(gal)			gallons per feet	1" ID 2" ID	4" ID 6" ID			
Pumping Rate of Pump:	(mL/min)			of water column:	0.041 0.163	0.653 1.469			
Pumping Rate of Pump:	(GPM)			1 gal = 3.7	85 L =3785 mL = 0.1	337 cubic ft.			
Minutes of Pumping:	(min)			Well Brushing M	ethod and Duratio	on:			
Total Volume Removed:	(gal)			Well Screen Surg	ing Method and I	Duration:			
EVACUATION INFORMATION Evacuation Method: Tubing Used:		Peristaltic	Grunfos 🔲 V	VaTerra	Other Pump	□			
		🗖							
Did well go dry?	Yes L	No	Water Qua	lity Meter Type:					
Did well go dry? Time 1 Parameter	Yes L	No 🛄 2	Water Qua	lity Meter Type: 4	5	6	7	8	9
Time 1				lity Meter Type:	5	6	7	8	9
Time 1 Parameter				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml)				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C)				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C) pH				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C)				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C) pH Conductance (mS/cm)				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C) pH Conductance (mS/cm) Dissolved Oxygen (mg/L)				4	5	6	7	8	9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C) pH Conductance (mS/cm) Dissolved Oxygen (mg/L) Turbidity (NTU) ORP (mV)		2	3	4			7		9
Time 1 Parameter Volume Purged (ml) Depth to Water (ft. TIC) Temperature (°C) pH Conductance (mS/cm) Dissolved Oxygen (mg/L) Turbidity (NTU)				4 4 13	5		7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     10		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     10       Parameter     1		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     1       Time     10       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     10       Parameter     10       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     1       Time     10       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     1       Time     10       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1		2	3	4			7		9
Time     1       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1       Dissolved Oxygen (mg/L)     1       Turbidity (NTU)     1       ORP (mV)     1       Time     10       Parameter     1       Volume Purged (ml)     1       Depth to Water (ft. TIC)     1       Temperature (°C)     1       pH     1       Conductance (mS/cm)     1		2	3	4			7		9



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### Water-Level and NAPL Thickness Measurement Procedures

Rev. #: 0

Rev Date: February 27, 2009

**Approval Signatures** 

sank shew Prepared by: Andrew Korik

Date: 2/27/09

Mur Reviewed by:

Michael Gefell (Technical Expert)

Date: 2/27/09

#### I. Scope and Application

Monitoring well water levels and thickness of non-aqueous phase liquids (NAPLs) will be determined, as appropriate, to develop groundwater elevation contour maps and to assess the presence or absence of NAPL in wells. This SOP applies to light and/or dense NAPLs (LNAPLs and DNAPLs, respectively). In addition, because this SOP describes water-level measurement from surveyed measurement points, this SOP can be followed, to obtain surface water level measurements from surveyed measurement points.

Fluid levels will be measured using an electric water-level probe and/or NAPL-water interface probe from established reference points. Reference points are surveyed, and are established at the highest point at the top of well riser, and will be based on mean sea level, or local/onsite datum. The Operating and Maintenance (O&M) Instruction Manual for the electric water level probe and/or and interface probe should be reviewed prior to commencing work for safe and accurate operation.

#### II. Personnel Qualifications

Individuals conducting fluid level measurements will have been trained in the proper use of the instruments, including their use for measuring fluid levels and the bottom depth of wells. In addition, ARCADIS field sampling personnel will have current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work. ARCADIS field personnel will also be compliant with client-specific training requirements, such as (but not limited to) LPS or other behavior-based training, and short-service employee restrictions.

#### III. Equipment List

The following materials, as required, shall be available during fluid level measurements.

- photoionization detector (PID)
- appropriate health and safety equipment, as specified in the site Health and Safety Plan (HASP)

- laboratory-type soap (Alconox or equivalent), methanol/hexane rinse, potable water, distilled water, and/or other equipment that may be needed for decontamination purposes
- electronic NAPL-water interface probe
- electronic water-level meter
- 6-foot engineer's rule
- portable containers
- plastic sheeting
- field logbook and/or personal digital assistant (PDA)
- indelible ink pen
- digital camera (optional, if allowed by site policy)

#### IV. Cautions

Electronic water-level probes and NAPL-water interface probes can sometimes produce falsepositive readings. For example, if the inside surface of the well has condensation above the water level, then an electronic water-level probe may produce a signal by contacting the side of the well rather than the true water level in the well. In addition, NAPL-water interface probes can sometimes indicate false positive signals when contacting a sediment layer on the bottom of a well. In contrast, a NAPL-water interface probe may produce a false-negative (no signal) if a floating layer of non-aqueous phase liquid (NAPL) is too thin, such as a film or sheen. To produce reliable data, the electronic water level probe and/or interface probe should be raised and lowered several times at the approximate depth where the instrument produces a tone indicating a fluid interface to verify consistent, repeatable results. In addition, a bottom-loading bailer should periodically be used to check for the presence of NAPLs rather than relying solely on the NAPL-water interface probe.

The graduated tape or cable with depth markings is designed to indicate the depth of the electronic sensor that detects the fluid interface, but not the depth of the bottom of the instrument. When using these devices to measure the total well depth, the additional length of the instrument below the electronic sensor must be added to the apparent well depth reading, as observed on the tape or cable of the instrument, to obtain the true total depth of the well. If the depth markings on the tape or cable are

worn or otherwise difficult to read, extra care must be taken in obtaining the depth readings.

#### V. Health and Safety Considerations

The HASP will be followed, as appropriate, to ensure the safety of field personnel. Access to wells may expose field personnel to hazardous materials such as contaminated groundwater or NAPL. Other potential hazards include stinging insects that may inhabit well heads, other biologic hazards, and potentially the use of sharp cutting tools (scissors, knife). Appropriate personal protective equipment (PPE) will be worn during these activities. Field personnel will thoroughly review client-specific health and safety requirements, which may preclude the use of fixed/folding-blade knives.

#### VI. Procedure

#### **Calibration Procedures**

If there is any uncertainty regarding the accuracy of the tape or cable associated with the electronic water-level probe or NAPL-water interface probe, it should be checked versus a standard length prior to use to assess if the tape or cable above the meter has been correctly calibrated by the manufacturer, and to identify evidence of tape or cable stretching, etc.

- Measure the lengths between markers on the cable with a 6-foot engineer's rule or a fiberglass engineer's tape. The tape or cable associated with the electronic water-level probe or NAPL-water interface probe should be checked for the length corresponding to the deepest total well depth to be monitored during the data collection event.
- 2. If the length designations on the tape or cable associated with the electronic water-level probe or NAPL-water interface probe are found to be incorrect, the probe will not be used until it is repaired by the manufacturer.
- 3. Record verification of this calibration process in field logbook or PDA.

#### **Measurement Procedures**

The detailed procedure for obtaining fluid level depth measurements is as follows. Field notes on logs will be treated as secured documentation and indelible ink will be used. As a general rule, the order of measuring should proceed from the least to most contaminated monitoring wells, based on available data.

- 1. Identify site and well number in field logbook using indelible ink, along with date, time, personnel, and weather conditions.
- Field personnel will avoid activities that may introduce contamination into monitoring wells. Activities such as dispensing gasoline into vehicles or generators should be accomplished well in advance of obtaining field measurements.
- 3. Don PPE as required by the HASP..
- 4. Clean the NAPL/water interface probe and cable in accordance with the appropriate cleaning procedures. Down-hole instrumentation should be cleaned prior to obtaining readings at the first monitoring well and upon completion of readings at each well.
- 5. Clean the NAPL/water level interface probe and cable with a soapy (Alconox) water rinse followed by a solvent rinse (if appropriate based on site-specific constituents of concern) an analyte-free water rinse Contain rinse water in a portable container that will be transferred to an on-site container.
- 6. Put clean plastic sheeting on the ground next to the well.
- 7. Unlock and open the well cover while standing upwind from the well. Place the well cap on the plastic sheeting.
- 8. Locate a measuring reference point on the well casing. If one is not found, initiate a reference point at the highest discernable point on the inner casing (or outer if an inner casing is not present) by notching with a hacksaw, or using an indelible marker. All down-hole measurements will be taken from the reference point established at each well on the inner casing (on the outer only if an inner casing is not present).
- 9. Measure to the nearest hundredth of a foot and record the height of the inner and outer casings (from reference point, as appropriate) to ground level.
- 10. Record the inside diameter of the well casing in the field log.
- 11. If an electronic water level probe is used to measure the water level, lower the probe until it emits a signal (tone and or light) indicating the top of the water surface. Gently raise and lower the instrument through this interface to confirm its depth. Measure and record the depth of the water surface, and the total well depth, to the nearest hundredth of a foot from the reference point at the top of

the well. Lower the probe to the bottom of the well to obtain a total depth measurement.

- 12. If a NAPL/water interface probe is being used to measure the depth and thickness of NAPL, lower the instrument until it emits a signal (tone and or light) indicating whether LNAPL is present. Continue to lower the NAPL/water level interface probe until it indicates the top of water. Lower the probe to the bottom of the well to obtain a total depth measurement. Note also of the depth indicating the bottom of water and top of DNAPL layer, if any, based on the signal emitted by the interface probe. At each fluid interface, gently raise and lower the instrument through each the interface to confirm its depth. Measure to the nearest hundredth of a foot and record the depth of each fluid interface, and the total well depth, from the reference point.
- 13. Clean the NAPL/water interface probe and cable in accordance with the appropriate cleaning procedures.
- 14. If using a bailer to confirm the presence/absence of NAPL, the bailer should either have been previously dedicated to the well, or be a new previously unused bailer.
- 15. Compare the depth of the well to previous records, and note any discrepancy.
- 16. Lock the well when all activities are completed.

#### VII. Waste Management

Decontamination fluids, PPE, and other disposable equipment will be properly stored on site in labeled containers and disposed of properly. Be certain that waste containers are properly labeled and documented in the field log book. Review appropriate waste management SOPs, which may be state- or client-specific.

#### VIII. Data Recording and Management

Fluid level measurement data will be recorded legibly on "write-in-the-rain" field notebook in indelible pen and/or a PDA. Field situations such as apparent well damage or suspected tampering, or other observations of conditions that may result in compromised data collection will be photographically documented where practicable.

### IX. Quality Assurance

As described in the detailed procedure, the electronic water-level meter and/or NAPLwater interface probe will be calibrated prior to use versus an engineer's rule to ensure accurate length demarcations on the tape or cable. Fluid interface measurements will be verified by gently raising and lowering the instrument through each interface to confirm repeatable results.

### X. References

No literature references are required for this SOP.



Imagine the result

Low-Flow Groundwater Purging and Sampling Procedures for Monitoring Wells

Rev. #: 3

Rev Date: March 9, 2009

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#### **Approval Signatures**

Date: 3/9/2009

Prepared by: Die S. Liper Reviewed by: Michael J. Leftle

(Technical Expert)

Date: 3/9/2009

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#### I. Scope and Application

Groundwater samples will be collected from monitoring wells to evaluate groundwater quality. The protocol presented in this standard operating procedure (SOP) describes the procedures to be used to purge monitoring wells and collect groundwater samples. This protocol has been developed in accordance with the United States Environmental Protection Agency (USEPA) Region I Low Stress (Low Flow) Purging and Sampling Procedures for the Collection of Groundwater Samples from Monitoring Wells (USEPA SOP No. GW0001; July 30, 1996). Both filtered and unfiltered groundwater samples may be collected using this low-flow sampling method. Filtered samples will be obtained using a 0.45-micron disposable filter. No wells will be sampled until well development has been performed in accordance with the procedures presented in the SOP titled Monitoring Well Development, unless that well has been sampled or developed within the prior 1-year time period. Groundwater samples will not be collected within 1 week following well development.

#### II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading groundwater sample collection activities should have a minimum of 2 years of previous groundwater sampling experience. ARCADIS personnel providing assistance to groundwater sample collection and associated activities should have a minimum of 6 months of related experience or an advanced degree in environmental sciences, engineering, hydrogeology, or geology.

The supervisor of the groundwater sampling team will have at least 1 year of previous supervised groundwater sampling experience.

Prior to mobilizing to the field, the groundwater sampling team should review and be thoroughly familiar with relevant site-specific documents including but not limited to the site work plan, field sampling plan, QAPP, HASP, and historical information. Additionally, the groundwater sampling team should review and be thoroughly familiar with documentation provided by equipment manufacturers for all equipment that will be used in the field prior to mobilization.

#### III. Equipment List

Specific to this activity, the following materials (or equivalent) will be available:

 Health and safety equipment (as required in the site Health and Safety Plan [HASP]).

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- Site Plan, well construction records, prior groundwater sampling records (if available).
- Sampling pump, which may consist of one or more of the following:
  - submersible pump (e.g., Grundfos Redi-Flo 2); -
  - peristaltic pump (e.g., ISCO Model 150); and/or
  - bladder pump (e.g., Marschalk System 1, QED Well Wizard, etc.). -
- Appropriate controller and power source for pump:
  - Submersible and peristaltic pumps require electric power from either a generator or a deep cell battery.
  - Submersible pumps such as Grundfos require a pump controller to run the pump
  - Bladder pumps require a pump controller and a gas source (e.g., air compressor or compressed  $N_2$  or  $CO_2$  gas cylinders).
- Teflon<sup>®</sup> tubing or Teflon<sup>®</sup>-lined polyethylene tubing of an appropriate size for the pump being used. For peristaltic pumps, dedicated Tygon<sup>®</sup> tubing (or other type as specified by the manufacturer) will also be used through the pump apparatus.
- Water-level probe (e.g., Solinist Model 101).
- Water-quality (temperature/pH/specific conductivity/ORP/turbidity/dissolved oxygen) meter and flow-through measurement cell. Several brands may be used, including:
  - YSI 6-Series Multi-Parameter Instrument;
  - Hydrolab Series 3 or Series 4a Multiprobe and Display; and/or \_
  - Horiba U-10 or U-22 Water Quality Monitoring System.
- Supplemental turbidity meter (e.g., Horiba U-10, Hach 2100P, LaMotte 2020). Turbidity measurements collected with multi-parameter meters have been shown to sometimes be unreliable due to fouling of the optic lens of the

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turbidity meter within the flow-through cell. A supplemental turbidity meter will be used to verify turbidity data during purging if such fouling is suspected. Note that industry improvements may eliminate the need for these supplemental measurements in the future.

- Appropriate water sample containers (supplied by the laboratory).
- Appropriate blanks (trip blank supplied by the laboratory).
- 0.45-micron disposable filters (if field filtering is required).
- Large glass mixing container (if sampling with a bailer).
- Teflon<sup>®</sup> stirring rod (if sampling with a bailer).
- Cleaning equipment.
- Groundwater sampling log (attached) or bound field logbook. •

Note that in the future, the client may acquire different makes/models of some of this equipment if the listed makes/models are no longer available, or as a result of general upgrades or additional equipment acquisitions. In the event that the client uses a different make/model of the equipment listed, the client will use an equivalent type of equipment (e.g., pumps, flow-through analytical cells) and note the specific make/model of the equipment used during a sampling event on the groundwater sampling log. In addition, should the client desire to change to a markedly different sampling methodology (e.g., discrete interval samplers, passive diffusion bags, or a yet to be developed technique), the client will submit a proposed SOP for the new methodology for USEPA approval prior to implementing such a change.

The maintenance requirements for the above equipment generally involve decontamination or periodic cleaning, battery charging, and proper storage, as specified by the manufacturer. For operational difficulties, the equipment will be serviced by a qualified technician.

#### IV. Cautions

If heavy precipitation occurs and no cover over the sampling area and monitoring well can be erected, sampling must be discontinued until adequate cover is provided. Rain water could contaminate groundwater samples.

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Do not use permanent marker or felt-tip pens for labels on sample container or sample coolers - use indelible ink. The permanent markers could introduce volatile constituents into the samples.

It may be necessary to field filter some parameters (e.g., metals) prior to collection, depending on preservation, analytical method, and project quality objectives.

Store and/or stage empty and full sample containers and coolers out of direct sunlight.

To mitigate potential cross-contamination, groundwater samples are to be collected in a pre-determined order from least impacted to impacted based on previous analytical data. If no analytical data are available, samples are collected in order of upgradient, then furthest downgradient to source area locations.

Be careful not to over-tighten lids with Teflon liners or septa (e.g., 40 mL vials). Overtightening can cause the glass to shatter or impair the integrity of the Teflon seal.

#### V. Health and Safety Considerations

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

If thunder or lighting is present, discontinue sampling and take cover until 30 minutes have passed after the last occurrence of thunder or lighting.

Use caution when removing well caps as well may be under pressure, cap can dislodge forcefully and cause injury.

Use caution when opening protective casing on stickup wells as wasps frequently nest inside the tops of the covers. Also watch for fire ant mounds near well pads when sampling in the south or western U.S.

#### VI. Procedure

Groundwater will be purged from the wells using an appropriate pump. Peristaltic pumps will initially be used to purge and sample all wells when applicable. If the depth to water is below the sampling range of a peristaltic pump (approximately 25 feet), submersible pumps or bladder pumps will be used provided the well is constructed with a casing diameter greater than or equal to 2 inches (the minimum well diameter capable of accommodating such pumps). Bladder pumps are preferred over peristaltic and submersible pumps if sampling of VOCs is required to prevent volatilization. For

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smaller diameter wells where the depth to water is below the sampling range of a peristaltic pump, alternative sampling methods (i.e., bailing or small diameter bladder pumps) will be used to purge and sample the groundwater. Purge water will be collected and containerized.

- 1. Calibrate field instruments according to manufacturer procedures for calibration.
- 2. Measure initial depth to groundwater prior to placement of pumps.
- 3. Prepare and install pump in well: For submersible and non-dedicated bladder pumps, decontaminate pump according to site decontamination procedures. Non-dedicated bladder pumps will require a new Teflon<sup>®</sup> bladder and attachment of an air line, sample discharge line, and safety cable prior to placement in the well. Attach the air line tubing to the air port on the top of the bladder pump. Attach the sample discharge tubing to the water port on the top of the bladder pump. Care should be taken not to reverse the air and discharge tubing lines during bladder pump set-up as this could result in bladder failure or rupture. Attach and secure a safety cable to the eyebolt on the top of bladder pump (if present, depending on pump model used). Slowly lower pump, safety cable, tubing, and electrical lines into the well to a depth corresponding to the approximate center of the saturated screen section of the well. Take care to avoid twisting and tangling of safety cable, tubing, and electrical lines while lowering pump into well; twisted and tangled lines could result in the pump becoming stuck in the well casing. Also, make sure to keep tubing and lines from touching the ground or other surfaces while introducing them into the well as this could lead to well contamination. If a peristaltic pump is being used, slowly lower the sampling tubing into the well to a depth corresponding to the approximate center of the saturated screen section of the well. The pump intake or sampling tube must be kept at least 2 feet above the bottom of the well to prevent mobilization of any sediment present in the bottom of the well.
- 4. Connect the pump to other equipment. If using a bladder pump, the discharge water line should be connected to the bottom inlet port on the flow-through cell connected to the water quality meter. Connect the air line to the pump controller output port. The pump controller should then be connected to a supply line from an air compressor or compressed gas cylinder using an appropriate regulator and air hose. Take care to tighten the regulator connector onto the gas cylinder (if used) to prevent leaks. Teflon tape may be used on the threads of the cylinder to provide a tighter seal. Once the air compressor or gas cylinder is connected to the pump controller, turn on the compressor or open the valve on the cylinder to begin the gas flow. Turn on the pump controller if an on/off switch

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is present and verify that all batteries are charged and fully operating before beginning to pump.

5. Measure the water level again with the pump in the well before starting the pump. Start pumping the well at 200 to 500 milliliters (mL) per minute (or at lower site-specific rate if specified). The pump rate should be adjusted to cause little or no water level drawdown in the well (less than 0.3 feet below the initial static depth to water measurement) and the water level should stabilize. The water level should be monitored every 3 to 5 minutes (or as appropriate, lower flow rates may require longer time between readings) during pumping if the well diameter is of sufficient size to allow such monitoring. Care should be taken not to break pump suction or cause entrainment of air in the sample. Record pumping rate adjustments and depths to water. If necessary, pumping rates should be reduced to the minimum capabilities of the pump to avoid pumping the well dry and/or to stabilize indicator parameters. A steady flow rate should be maintained to the extent practicable. Groundwater sampling records from previous sampling events (if available) should be reviewed prior to mobilization to estimate the optimum pumping rate and anticipated drawdown for the well in order to more efficiently reach a stabilized pumping condition.

If the recharge rate of the well is very low, alternative purging techniques should be used, which will vary based on the well construction and screen position. For wells screened across the water table, the well should be pumped dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. For wells screened entirely below the water table, the well should be pumped until a stabilized level (which may be below the maximum displacement goal of 0.3 feet) can be maintained and monitoring for stabilization of field indicator parameters can commence. If a lower stabilization level cannot be maintained, the well should be pumped until the drawdown is at a level slightly higher than the bentonite seal above the well screen. Sampling should commence after one well volume has been removed and the well has recovered sufficiently to permit collection of samples.

During purging, monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, pH, etc.) every 3 to 5 minutes (or as appropriate). Field indicator parameters will be measured using a flow-through analytical cell or a clean container such as a glass beaker. Record field indicator parameters on the groundwater sampling log. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the specific conductance and temperature values remain within 3%, and pH remains within 0.1 units for three consecutive readings collected at 3- to 5-minute intervals (or

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other appropriate interval, alternate stabilization goals may exist in different geographic regions, consult the site-specific Work Plan for stabilization criteria). If the field indicator parameters do not stabilize within 1 hour of the start of purging, but the groundwater turbidity is below the goal of 50 NTU and the values for all other parameters are within 10%, the well can be sampled. If the parameters have stabilized but the turbidity is not in the range of the 50 NTU goal, the pump flow rate should be decreased to a minimum rate of 100 mL/min to reduce turbidity levels as low as possible. If dissolved oxygen values are not within acceptable range for the temperature of groundwater (Attachment 1), then check for and remove air bubbles on probe or in tubing. If the dissolved oxygen value is 0.00 or less, then the meter should be serviced and re-calibrated.

During extreme weather conditions, stabilization of field indicator parameters may be difficult to obtain. Modifications to the sampling procedures to alleviate these conditions (e.g., measuring the water temperature in the well adjacent to the pump intake) will be documented in the field notes. If other field conditions exist that preclude stabilization of certain parameters, an explanation of why the parameters did not stabilize will also be documented in the field logbook.

- 6. Complete the sample label and cover the label with clear packing tape to secure the label onto the container.
- 7. After the indicator parameters have stabilized, collect groundwater samples by diverting flow out of the unfiltered discharge tubing into the appropriate labeled sample container. If a flow-through analytical cell is being used to measure field parameters, the flow-through cell should be disconnected after stabilization of the field indicator parameters and prior to groundwater sample collection. Under no circumstances should analytical samples be collected from the discharge of the flow-through cell. When the container is full, tightly screw on the cap. Samples should be collected in the following order: VOCs, TOC, SVOCs, metals and cyanide, and others (or other order as defined in the site-specific Work Plan).
- 8. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Install an in-line, disposable 0.45-micron particle filter on the discharge tubing after the appropriate unfiltered groundwater sample has been collected. Continue to run the pump until an initial volume of "flush" water has been run through the filter in accordance with the manufacturer's directions (generally 100 to 300 mL). Collect filtered groundwater sample by diverting flow out of the filter into the appropriately labeled sample container. When the container is full, tightly screw on the cap.

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- 9. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.
- 10. Record on the groundwater sampling log or bound field logbook the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, and the presence or lack of odors or sheens), and the values of the stabilized field indicator parameters as measured during the final reading during purging (Attachment 2 – Example Sampling Log).
- 11. Turn off the pump and air compressor or close the gas cylinder valve if using a bladder pump set-up. Slowly remove the pump, tubing, lines, and safety cable from the well. Do not allow the tubing or lines to touch the ground or any other surfaces which could contaminate them.
- 12. If tubing is to be dedicated to a well, it should be folded to a length that will allow the well to be capped and also facilitate retrieval of the tubing during later sampling events. A length of rope or string should be used to tie the tubing to the well cap. Alternatively, if tubing and safety line are to be saved and reused for sampling the well at a later date they may be coiled neatly and placed in a clean plastic bag that is clearly labeled with the well ID. Make sure the bag is tightly sealed before placing it in storage.
- 13. Secure the well and properly dispose of personal protective equipment (PPE) and disposable equipment.
- 14. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody.
- 15. Complete decontamination procedures for flow-through analytical cell and submersible or bladder pump, as appropriate.
- 16. At the end of the day, perform calibration check of field instruments.

If it is not technically feasible to use the low-flow sampling method, purging and sampling of monitoring wells may be conducted using the bailer method as outlined below:

- 1. Don appropriate PPE (as required by the HASP).
- 2. Place plastic sheeting around the well.
- 3. Clean sampling equipment.

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- 4. Open the well cover while standing upwind of the well. Remove well cap and place on the plastic sheeting. Insert PID probe approximately 4 to 6 inches into the casing or the well headspace and cover with gloved hand. Record the PID reading in the field log. If the well headspace reading is less than 5 PID units, proceed; if the headspace reading is greater than 5 PID units, screen the air within the breathing zone. If the breathing zone reading is less than 5 PID units, proceed. If the PID reading in the breathing zone is above 5 PID units, move upwind from well for 5 minutes to allow the volatiles to dissipate. Repeat the breathing zone test. If the reading is still above 5 PID units, don appropriate respiratory protection in accordance with the requirements of the HASP. Record all PID readings. For wells that are part of the regular weekly monitoring program and prior PID measurements have not resulted in a breathing zone reading above 5 PID units, PID weasurements will be taken monthly.
- 5. Measure the depth to water and determine depth of well by examining drilling log data or by direct measurement. Calculate the volume of water in the well (in gallons) by using the length of the water column (in feet), multiplying by 0.163 for a 2-inch well or by 0.653 for a 4-inch well. For other well diameters, use the formula:

Volume (in gallons) = • TIMES well radius (in feet) squared TIMES length of water column (in feet) TIMES 7.481 (gallons per cubic foot)

- 6. Measure a length of rope or twine at least 10 feet greater than the total depth of the well. Secure one end of the rope to the well casing and secure the other end to the bailer. Test the knots and make sure the rope will not loosen. Check bailers so that all parts are intact and will not be lost in the well.
- 7. Lower bailer into well and remove one well volume of water. Contain all water in appropriate containers.
- Monitor the field indicator parameters (e.g., turbidity, temperature, specific conductance, and pH). Measure field indicator parameters using a clean container such as a glass beaker or sampling cups provided with the instrument. Record field indicator parameters on the groundwater sampling log.
- 9. Repeat Steps 7 and 8 until three or four well volumes have been removed. Examine the field indicator parameter data to determine if the parameters have stabilized. The well is considered stabilized and ready for sample collection when turbidity values remain within 10% (or within 1 NTU if the turbidity reading is less than 10 NTU), the specific conductance and temperature values remain

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within 3%, and pH remains within 0.1 units for three consecutive readings collected once per well volume removed.

- 10. If the field indicator parameters have not stabilized, remove a maximum of five well volumes prior to sample collection. Alternatively, five well volumes may be removed without measuring the field indicator parameters.
- 11. If the recharge rate of the well is very low, wells screened across the water table may be bailed dry and sampling should commence as soon as the volume in the well has recovered sufficiently to permit collection of samples. For wells screened entirely below the water table, the well should only be bailed down to a level slightly higher than the bentonite seal above the well screen. The well should not be bailed completely dry, to maintain the integrity of the seal. Sampling should commence as soon as the well volume has recovered sufficiently to permit sample collection.
- 12. Following purging, allow water level in well to recharge to a sufficient level to permit sample collection.
- 13. Complete the sample label and cover the label with clear packing tape to secure the label onto the container.
- 14. Slowly lower the bailer into the screened portion of the well and carefully retrieve a filled bailer from the well causing minimal disturbance to the water and any sediment in the well.
- 15. The sample collection order (as appropriate) will be as follows:
  - a. VOCs;
  - b TOC;
  - c. SVOCs;
  - d. metals and cyanide; and
  - e. others.
- When sampling for volatiles, collect water samples directly from the bailer into 40-mL vials with Teflon<sup>®</sup>-lined septa.

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- 17. For other analytical samples, remove the cap from the large glass mixing container and slowly empty the bailer into the large glass mixing container. The sample for dissolved metals and/or filtered PCBs should either be placed directly from the bailer into a pressure filter apparatus or pumped directly from the bailer with a peristaltic pump, through an in-line filter, into the pre-preserved sample bottle.
- 18. Continue collecting samples until the mixing container contains a sufficient volume for all laboratory samples.
- 19. Mix the entire sample volume with the Teflon<sup>®</sup> stirring rod and transfer the appropriate volume into the laboratory jar(s). Secure the sample jar cap(s) tightly.
- 20. If sampling for total and filtered metals and/or PCBs, a filtered and unfiltered sample will be collected. Sample filtration for the filtered sample will be performed in the field using a peristaltic pump prior to preservation. Install new medical-grade silicone tubing in the pump head. Place new Teflon<sup>®</sup> tubing into the sample mixing container and attach to the intake side of pump tubing. Attach (clamp) a new 0.45-micron filter (note the filter flow direction). Turn the pump on and dispense the filtered liquid directly into the laboratory sample bottles.
- 21. Secure with packing material and store at 4°C in an insulated transport container provided by the laboratory.
- 22. After sample containers have been filled, remove one additional volume of groundwater. Measure the pH, temperature, turbidity, and conductivity. Record on the groundwater sampling log or bound field logbook the time sampling procedures were completed, any pertinent observations of the sample (e.g., physical appearance, and the presence or lack of odors or sheens), and the values of the field indicator parameters.
- 23. Remove bailer from well, secure well, and properly dispose of PPE and disposable equipment.
- 24. If a bailer is to be dedicated to a well, it should be secured inside the well above the water table, if possible. Dedicated bailers should be tied to the well cap so that inadvertent loss of the bailer will not occur when the well is opened.
- 25. Complete the procedures for packaging, shipping, and handling with associated chain-of-custody.

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#### VII. Waste Management

Materials generated during groundwater sampling activities, including disposable equipment, will be placed in appropriate containers. Containerized waste will be disposed of by the client consistent with the procedures identified in the HASP.

#### VIII. Data Recording and Management

Initial field logs and chain-of-custody records will be transmitted to the ARCADIS PM at the end of each day unless otherwise directed by the PM. The groundwater team leader retains copies of the groundwater sampling logs.

#### IX. Quality Assurance

In addition to the quality control samples to be collected in accordance with this SOP, the following quality control procedures should be observed in the field:

- Collect samples from monitoring wells in order of increasing concentration, to the extent known based on review of historical site information if available.
- Equipment blanks should include the pump and tubing (if using disposable tubing) or the pump only (if using tubing dedicated to each well).
- Collect equipment blanks after wells with higher concentrations (if known) have been sampled.
- Operate all monitoring instrumentation in accordance with manufacturer's instructions and calibration procedures. Calibrate instruments at the beginning of each day and verify the calibration at the end of each day. Record all calibration activities in the field notebook.
- Clean all groundwater sampling equipment prior to use in the first well and after each subsequent well using procedures for equipment decontamination.

## X. References

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## Attachment 1

## Groundwater Sampling Log



Low-Flow Groundwater Sampling Log

Project										
Project Number			Site Location Well ID							
Date		Sampled By								
Sampling Time		Recorded By								
Weather				Coded Replica	ate No.					
Instrument Ide						0				
Water Quality	Meter(s)					Serial #				
Casing Materia	al			Purge	Method	_				
Cooing Diameter				Screen	Interval (ft br	np) Top	Bottom			
Sounded Denth (ft hmm)				Pump Intake Depth (ft bmp)						
Depth to Wate	r (ft bmp)						Finish			
				Field Parameter	Measuremen	ts During Purging				
Time	Minutes	Flow Rate	Volume	Temp	рН	Conductivity (umhos or	ORP	DO	Turbidity	Depth to
Time	Elasped	(mL/min)	Purged	(°C)	(s.u.)	(unnos or mS/cm) <sup>1)</sup>	(mV)	(mg/L)	(NTU)	Water (ft bmp)
						+ +				
Collected Sam	ple Condition		Color	•	Odor	•		Appearance_	•	
Parameter			Container			No.		–	Preservative	
		-			-			_		
		-			-			_		
PID Reading		_			-			_		
_			-							
Comments										

1) Circle one unit type

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#### Attachment 2

#### Oxygen Solubility in Fresh Water

Temperature	Dissolved Oxygen
(degrees C)	(mg/L)
0	14.6
1	14.19
2	13.81
3	13.44
4	13.09
5	12.75
6	12.43
7	12.12
8	11.83
9	11.55
10	11.27
11	11.01
12	10.76
13	10.52
14	10.29
15	10.07
16	9.85
17	9.65
18	9.45
19	9.26
20	9.07
21	8.9
22	8.72
23	8.56
24	8.4
25	8.24
26	8.09
27	7.95
28	7.81
29	7.67
30	7.54
31	7.41
32	7.28
33	7.16
34	7.05
35	6.93

Reference: Vesilind, P.A., *Introduction to Environmental Engineering*, PWS Publishing Company, Boston, 468 pages (1996).

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# Standard Operating Procedure for LNAPL Sample Collection and Shipping

Rev. #: 1.0

Rev Date: March 26, 2009

**Approval Signatures** 

HA Mi Kalluk (Trika Nelson-Kalmes)

Date: 3/26/09

Date: 3/26/09

Prepared by:

BAK

Reviewed by:

(Brad Koons)

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SOP: LNAPL Sample Collection and Shipping 2 Rev. #: 1.0 | Rev Date: April 15, 2009

#### I. Scope and Application

Subsurface fluid sample collection is often required to characterize Light Nonaqueous Phase Liquid (LNAPL) properties at petroleum-impacted sites. The subsurface fluids (groundwater and separate-phase petroleum product) are submitted to an analytical laboratory(s) for specialized physical testing (e.g., density, viscosity, interfacial tension) and/or chemical speciation testing. It is important to note that the physical parameters are temperature sensitive. Therefore, the laboratory should be directed to analyze the samples at representative subsurface fluid temperatures. The fluid data are used to support site-specific LNAPL mobility calculations and development of the LNAPL site conceptual model.

This SOP does not address details of drilling method selection; soil description; or laboratory analysis. Refer to other ARCADIS SOPs and the project work plan, as appropriate.

#### II. Personnel Qualifications

ARCADIS personnel overseeing, directing, or supervising LNAPL fluid collection shall have previous related experience (minimum of 2 years) collecting fluid samples from wells and shall be trained in shipping of hazardous materials.

#### III. Equipment List

- personal protective equipment (PPE), items specified by the site Health and Safety Plan (HASP), and first aid kit;
- measuring tape;
- scissors;
- indelible ink pens;
- site map;
- contact names and numbers;
- well lock keys;
- logbook;
- interface probe;
- cleaning equipment/supplies, including deionized (DI) water and LiquiNox or equivalent;
- plastic sheeting;
- sampling containers;
- bailers, rope, and bailer retrieval device;
- buckets;
- bubble wrap and Styrofoam peanuts;
- duct tape and clear packaging tape;
- shippable cooler or sturdy box;
- shipping labels;
- chain of custody forms;
- garbage bags; and
- drum bung wrench.

#### IV. Cautions

Please refer to the Site specific HASP and JSAs for the Site.

#### V. Health and Safety Considerations

Field activities associated with collection of nonaqueous phase liquids and water will be performed in accordance with a site specific HASP, a copy of which will be present on site during such activities. The field staff must be made aware of hazardous substances that may be present in the groundwater and nonaqueous phase liquids and understand the associated health hazards.

#### VI. Fluid Sample Collection Procedure

- 1. Measuring the static water level: Proper PPE must be worn (i.e. gloves, safety glasses, steel-toed boots, etc.). Remove cap from well and deploy the oil/LNAPL and water interface probe into the well. Measure the static LNAPL and water levels in each well before sampling. Decontaminate the interface probe using LiquiNox (or equivalent) and DI water between well measurements. Read fluid level measurements to the nearest 0.01 foot on the north side, top of casing. Use the same electronic oil and water interface probe for all wells. Make sure to record all depths to product (DTP) and depths to water (DTW) in the field book. Depending on the probe, it will make different sounds for water and oil/LNAPL.
- 2. Collecting LNAPL and groundwater samples: Dedicated bailer and rope must be used for each well. Make sure to sample in the same order that water and LNAPL levels were collected to avoid any cross contamination. Collect the LNAPL sample by slowly lowering the bailer into the LNAPL, but not into the water. Pull the bailer out of the well. If both water and LNAPL are present, allow the liquids to separate. Collect the groundwater sample by lowering the bailer. Use a bottom emptying device to decant (drain) the appropriate amount of LNAPL or water into the appropriate container(s), as described below. Drain off remaining, unneeded liquids into a 5 gallon "waste" bucket. Record the amount of LNAPL bailed from each well in the logbook. The required sample volumes and containers, indicated below, are dependent upon the laboratory analyses to be performed.
  - a. Fluid Properties Analysis: Requires 250 mL (minimum) of site groundwater and 250 mL (minimum) of LNAPL. The groundwater and LNAPL must be separated and placed into separate 1-liter glass containers.

- b. Water/LNAPL Relative Permeability: Requires 1 to 2 liters (minimum) of field water and 1 liter (minimum) of LNAPL, placed in up to three 1-liter glass containers. It is preferable that LNAPL and field water are separated into separate sample containers.
- Use waterproof labels for the containers and permanent waterproof marking devices for labeling. Labels are to include unique sample IDs, collection date and time, sampler initials, and lab analyses to be performed. These samples **DO NOT** need to be chemically preserved or shipped on ice.
- 4. Once sampling is complete, put the cap back on the well, close, and secure it as necessary. Personal protective equipment (such as gloves and disposable clothing) and other disposable equipment resulting from cleaning procedures and LNAPL and water sampling/handling activities (such as paper towels, rope, and bailers) will be placed in plastic garbage bags. Disposable PPE and equipment should not be re-used. Dispose of any excess water/LNAPL from the well into a 55-gallon drum or on site poly tank for proper disposal at a later date. Follow the procedures outlined in the Waste Management section below for further waste handling.

#### VII. Sample Shipping Procedure

The United States Department of Transportation (DOT) hazardous shipping guidelines must be followed when shipping LNAPL. Hazardous samples being shipped by ARCADIS staff must have completed current training through ARCADIS for DOT training for hazardous material shipping. A shipping determination form must be completed for all samples being shipped along with following all ARCADIS and DOT shipping guidelines. All forms and guidelines can be found online at <a href="http://team/sites/hazmat/default.aspx">http://team/sites/hazmat/default.aspx</a>. If there are additional questions contact Sam Moyers (ARCADIS H&S).

#### VIII. Waste Management

The plastic garbage bags containing disposable PPE and equipment will be transferred into appropriately labeled 55-gallon drums or disposed of in a designated debris box for disposal. All decontamination and well water will be placed in separate sealed 55-gallon steel drums and stored in a secured area. Once full, the material will be analyzed to determine the appropriate disposal method.

SOP: LNAPL Sample Collection and Shipping 5 Rev. #: 1.0 | Rev Date: April 15, 2009

#### IX. Data Recording and Management

The supervising geologist/engineer will be responsible for documenting sampling events using a logbook to record all relevant information in a clear and concise format. The sampling event record shall include:

- name and location of project;
- project number, client, and site location;
- names of Contractor, Contractor personnel, inspectors, and other people onsite;
- weather conditions;
- depth to groundwater and depth to LNAPL;
- type of sampling method;
- start and finish dates and times of sampling;
- volume of groundwater bailed and sampled;
- LNAPL as measured in a graduated cylinder and sampled; and
- photo document the LNAPL and cooler packaging.

#### X. Quality Assurance

Equipment will be cleaned prior to use onsite, between each sampling location, and prior to leaving the site.

Review bottle labels and the COC prior to shipping to ensure everything is labeled and documented correctly.

#### XI. References

PTS Laboratories, 2009. www.ptslabs.com



Imagine the result

Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation

Rev. #: 2

Rev Date: August 19, 2009

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008

### **Approval Signatures**

Prepared by:

Date: Mitch Wacksman and Michael Strikler

Reviewed by:

(Technical Expert) Christopher Lutes

Date: 11/14/08

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SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008

#### I. Scope and Application

This document describes the procedures to collect subsurface soil-gas samples for the analysis of volatile organic compounds (VOCs) by United States Environmental Protection Agency (USEPA) Method TO-15 (TO-15). The TO-15 method uses a 6-liter SUMMA® passivated stainless-steel canister. An evacuated 6-liter SUMMA® canister (<28 inches of mercury [Hg]) will provide a recoverable whole-gas sample of approximately 5.5 liters when allowed to fill to a vacuum of 2 inches of Hg. The whole-air sample will be analyzed for VOCs using a quadrupole or ion-trap gas chromatograph/mass spectrometer (GC/MS) system to provide compound detection limits of 0.5 parts per billion volume (ppbv).

The following sections list the necessary equipment and provide detailed instructions for the installation of soil-gas probes (using direct-push, hollow-stem and hand auger technologies) and the collection of soil-gas samples for VOC analysis.

This SOP should be reviewed prior to work plan preparation to ensure its compliance with specific regulatory and/or client requirements for subsurface soil-gas sampling.

#### II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, site-specific training, first-aid, and cardiopulmonary resuscitation (CPR), as needed. ARCADIS field sampling personnel will be well versed in the relevant standard operating procedures (SOPs) and possess the required skills and experience necessary to successfully complete the desired field work. ARCADIS personnel responsible for leading subsurface soil-gas sample collection activities must have previous subsurface soil-gas sampling experience.

#### III. Equipment List

The equipment required to install a soil vapor probe is presented below:

- Appropriate PPE (as required by the Health and Safety Plan);
- Appropriate drill rig to reach necessary sample depth (hollow-stem auger, direct-push rig, hand auger, etc.)

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008

- Direct-push rig (e.g., PowerProbe<sup>™</sup>) equipped with interconnecting 4foot lengths of 1.25-inch-diameter steel rods; or
- Hollow-stem auger drill rig with interconnecting augers. The inner diameter of typical augers range from 2.25-inches to 7.75 inches.
- Hand auger equipped with the necessary lengths of shaft extenders to reach the desired depth;
- Commercially available stainless steel sample screens
- Expendable points (one per sample);
- Expendable point holder, and appropriate twist-to-lock (or Swagelok®) connector;
- Stainless steel, brass or Teflon® ball valve or needle valve;
- Photoionization Detector (with a lamp of 11.7 eV);
- ¼-inch inside diameter (ID) tubing (Teflon®, Teflon®-lined polyethylene, or PEEK);
- Commercially available clean sand or play sand;
- Non-coated bentonite (dry chips and wet slurry);
- Down-hole measuring device; and
- Traffic-rated well cover (for permanent installations)
- Kneeling pad

The equipment required for soil-gas sample collection is presented below:

- Stainless steel SUMMA® canisters (order at least one extra, if feasible);
- Flow controllers with in-line particulate filters and vacuum gauges; flow controllers are pre-calibrated to specified sample duration (e.g., 30 minutes, 8 hours, 24 hours) or flow rate (e.g., 200 milliliters per minute [mL/min]);

confirm with the laboratory that the flow controller comes with an in-line particulate filter and pressure gauge (order at least one extra, if feasible),

- flow controllers pre-calibrated to the appropriate duplicate sampling time (typically double the standard sample time);
- Slower flow rates will likely be required in silty or clay soils.
- 1/4-inch ID tubing (Teflon® or Teflon®-lined polyethylene);
- Twist-to-lock or Swagelok® fittings;
- Stainless steel "T" fitting (if collecting duplicate [i.e., split] samples);
- Portable vacuum pump capable of producing very low flow rates (e.g., 100 to 200 mL/min) with vacuum gauge;
- Rotameter or an electric flow sensor if vacuum pump does not have a flow gauge;
- Tracer gas source (e.g., helium);
- Tracer gas detector;
- PID;
- Appropriate-sized open-end or flare-nut wrench (typically 9/16-inch + ½"); flare-nut wrenches as opposed to open-end wrenches can reduce the risk of the wrench slipping off of the fitting while tightening (and possibly causing hand injury or damage to the sampling train
- Chain-of-custody (COC) form;
- Sample collection log (attached); and
- Field notebook.

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008

#### IV. Cautions

Sampling personnel should not handle hazardous substances (such as gasoline), permanent marking pens, wear/apply fragrances, wear dry-cleaned clothing, or smoke cigarettes/cigars before and/or during the sampling event.

Care should be taken to ensure that the flow controller is pre-calibrated to the proper sample collection time (confirm with laboratory) and is capable of being transported without damage to the calibration. Sample integrity is maintained if the sampling event is shorter than the target duration, but sample integrity can be compromised if the event is extended to the point that the canister reaches atmospheric pressure.

Care must be taken to properly seal around the vapor probe at ground surface and to fully tighten (but not over-tighten) fittings to prevent leakage of atmosphere into the soil vapor probe and sampling train during purging and sampling. Temporary sampling points are to be sealed at the surface using hydrated bentonite. Permanent points are sealed at the surface using hydraulic cement powder.

#### V. Health and Safety Considerations

Field sampling equipment must be carefully handled to minimize the potential for injury and the spread of hazardous substances. For subsurface vapor probe installation, drilling with a direct-push or hollow-stem drilling rig should be done only by personnel with prior experience using such a piece of equipment.

#### VI. Procedure

#### Soil-Gas Steel Rod Monitoring Point Installation

- 1. Advance an assembly, consisting of interconnected lengths of decontaminated 1.25-inch-diameter steel drive rods, affixed with an expendable point holder and expendable point at the downhole end to the bottom of the desired sampling interval.
- 2. Cut a length of sample collection tubing slightly longer (e.g., 1 to 2 feet) than the collection depth. Attach a twist-to-lock connector to one end of the sample collection tubing and lower the twist-to-lock connector and attached tubing through the drive rods. Thread the twist-to-lock connector into the expendable point holder by twisting counterclockwise.

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- 3. Hydraulically retract the sampling assembly approximately 6 inches or more if needed, allowing the expendable point to fall off and creating a void in the subsurface for soil-gas sample collection.
- 4. Fill annular space between the steel drive rod and the borehole wall (if any) with bentonite. Typically, only a bentonite surface seal is needed since there is no annular space between the steel drive rods and the borehole wall.
- 5. Proceed to soil-gas sample collection.

#### Soil-Gas Hollow-Stem Auger Monitoring Point Installation

- 1. Advance boring just past the bottom of deepest sampling interval.
- 2. Fill the boring with sand to the deepest sampling interval.

3. Cut a length of sample collection tubing slighty longer (e.g., 4 to 5 feet) than the collections depth. Attach a stainless steel sample screen to one end of the sample collection tubing and lower the screen and attached tubing down the middle of the augers.

4. Assure that the sample screen has reached the bottom of the boring and record this depth.

5. Being simultaneously filling in the area around the sample screen with clean sand and retracting the augers. Sand should be introduced to cover the sample screen then to extend above the screen per work plan instructions.

6. With the proper sand pack in place, slowly add a layer of dry bentonite chips to prevent moisture from the hydrated bentonite slurry from reaching the sampling point.

7. With the dry bentonite chips in place, slowy pour hydrated bentonite down the augers while simultaneously retracting the augers.

8. Seal the boring per work plan instructions (typically hydrated bentonite chips and concrete).

9. Affix a Swagelok® fitting and valve to the end of the tubing.

10. Properly label the sample tubing and valve with a permanent label to designate the sample number and screen depth.

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008 7

11. All soil-gas points should be allowed to equilibrate for a minimum of 48 hours before proceeding to soil-gas sample collection.

#### Hand Auger Monitoring Point Installation

The procedures for hand auger monitoring point installation are very similar to the hollow-stem auger procedure. Once the boring has been cleared to just past its final depth, the entire hand auger should be removed from the boring. Monitoring point installation should then commence as described for the hollow-stem auger installation.

#### **Soil-Gas Sample Collection**

#### Preparation of SUMMA®-Type Canister and Collection of Sample

- 1. Record the following information in the field notebook, if appropriate (contact the local airport or other suitable information source [e.g., site-specific measurements, weatherunderground.com] to obtain the information):
  - a. wind speed and direction;
  - b. ambient temperature;
  - c. barometric pressure; and
  - d. relative humidity.
- 2. Connect a short piece of Teflon tubing to the sub-slab sampling port using a twist-to-lock fitting.
- 3. Connect a portable vacuum pump to the sample tubing. Purge 1 to 2 (target 1.5) volumes of air from the vapor probe and sampling line using a portable pump [purge volume = 1.5 Pi r2h] at a rate of approximately 100 mL/min. Measure organic vapor levels with the PID. Lower flow rates may be necessary in silt or clay to avoid excessive vacuum. Vacuum is >136 inches of water column are clearly excessive. Other sources site a cutoff of >10 inches of water column.
- 4. Check the seal established around the soil vapor probe by using a tracer gas (e.g., helium) or other method established in the state guidance documents.

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[Note: Refer to SOP "Administering Tracer Gas," adapted from NYSDOH 2005, for procedures on tracer gas use.]

- 5. Remove the brass or stainless steel plug from the SUMMA® canister and connect the flow controller with in-line particulate filter and vacuum gauge to the SUMMA® canister. Do not open the valve on the SUMMA® canister. Record in the field notebook and COC form the flow controller number with the appropriate SUMMA® canister number.
- 6. Connect the Teflon sample collection tubing to the flow controller and the SUMMA® canister valve. Record in the field notebook the time sampling began and the canister pressure.
- 7. Connect the other end of the polyethylene tubing to the sub-slab sampling port.
- 8. Open the SUMMA® canister valves. Record in the field notebook the time sampling began and the canister pressure.
- 9. Take a photograph of the SUMMA® canister and surrounding area.

#### Termination of Sample Collection

- 1. Arrive at the SUMMA® canister location at least 10 to 15 minutes prior to the end of the required sampling interval (e.g., 30 to 60 minutes).
- 2. Record the final vacuum pressure. Stop collecting the sample by closing the SUMMA® canister valves. The canister should have a minimum amount of vacuum (approximately 2 inches of Hg or slightly greater).
- 3. Record the date and local time (24-hour basis) of valve closing in the field notebook, sample collection log, and COC form.
- 4. Remove the particulate filter and flow controller from the SUMMA® canister, reinstall the brass plug on the canister fitting, and tighten with the appropriate wrench.
- 5. Package the canister and flow controller in the shipping container supplied by the laboratory for return shipment to the laboratory. The SUMMA® canister does not require preservation with ice or refrigeration during shipment.

- SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008
- 6. Complete the appropriate forms and sample labels as directed by the laboratory (e.g., affix card with a string).

Complete the COC form and place the requisite copies in a shipping container. Close the shipping container and affix a custody seal to the container closure. Ship the container to the laboratory via overnight carrier (e.g., Federal Express) for analysis.

#### **Soil-Gas Monitoring Point Abandonment**

Once the soil-gas samples have been collected, the soil-gas monitoring points will be abandoned by removing the drive rods and filling the resulting hole with bentonite.

#### VII. Waste Management

Field personnel will collect and remove all investigation-derived waste materials (including disposable equipment) for proper disposal.

#### VIII. Data Recording and Management

Measurements will be recorded in the field notebook at the time of measurement with notations of the project name, sample date, sample start and finish time, sample location (e.g., GPS coordinates, distance from permanent structure), canister serial number, flow controller serial number, initial vacuum reading, and final pressure reading. Field sampling logs and COC records will be transmitted to the Project Manager.

#### IX. Quality Assurance

Soil-gas sample analysis will be performed using USEPA TO-15 methodology. This method uses a quadrupole or ion-trap GC/MS with a capillary column to provide optimum detection limits. The GC/MS system requires a 1-liter gas sample (which can easily be recovered from a 6-liter canister) to provide a 0.5-ppbv detection limit. The 6-liter canister also provides several additional 1-liter samples in case subsequent reanalyses or dilutions are required. This system also offers the advantage of the GC/MS detector, which confirms the identity of detected compounds by evaluating their mass spectra.

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 – 10 Single Port, Direct Push, Hollow-Stem and Hand Auger Installation Rev. #: 1 | Rev Date: November 14, 2008

All analytical results will be reported in units of • g/m3.

#### X. References

New York State Department of Health (NYSDOH). 2005. DRAFT "Guidance for Evaluating Soil Vapor Intrusion in the State of New York" February 23, 2005.

SOP: Subsurface Soil-Gas Sampling and Analysis Using USEPA Method TO-15 1 Rev. #: 0 | Rev Date: March 30, 2006

<b>ARCADIS</b>	Soil-Gas Sample Collection Log			
Infrastructure, environment, facilities		Sample ID:		
Client:		Date/Day:		
Project:		Weather:		
Location:		Temperature:		
Project #:		Wind Speed/Direction:		
Samplers:		Subcontractor:		
Logged By:		Equipment:		
Coordinates:		Moisture Content of Sampling Zone (circle one):	Dry / Moist	
Sampling Depth:		Approximate Purge Volume:		
Time of Collection:		Background PID Ambient Air Reading:		

#### Nearby Groundwater Monitoring Wells/Water Levels:

Well ID	Depth to Groundwater (ft)

#### **SUMMA Canister Information:**

Size (circle one):	1 L	6 L
Canister ID:		

Flow Controller ID:

Tracer Gas Information (if applicable)

Tracer Gas:

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Canister Pressure (inches Hg):		
Reported By Laboratory	Measured Prior to Sample Collection	Measured Following Sample Collection

Tracer Gas Concentration (if applicable):				
Measured in Purge Effluent	Measured in 'Concentrated' Area Prior to Sample Collection	Measured in 'Concentrated' Area Following Sample Collection		
General Observations/Notes:				

#### General Observations/Notes:

#### Approximating One-Well Volume (for purging):

When using 1<sup>1</sup>/<sub>4</sub>-inch "Dummy Point" and a 6-inch sampling interval, the sampling space will have a volume of approximately 150 mL. Each foot of <sup>1</sup>/<sub>4</sub>-inch tubing will have a volume of approximately 10 mL.



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## Ambient Air Sampling and Analysis Using USEPA Method TO-15

Rev. #: 1

Rev Date: March 13, 2009

SOP: Ambient Air Sampling and Analysis Using USEPA Method TO-15 1 Rev. #: 1 | Rev Date: March 13, 2009

**Approval Signatures** 

adine Weinberg Date: \_ Prepared by: 3/13/09

Nadine Weinberg

Reviewed by:

Date: 3/13/09

Christopher Lutes (Technical Expert)

#### I. Scope and Application

This standard operating procedure (SOP) describes the procedures to collect ambient air samples for the analysis of volatile organic compounds (VOCs) using United States Environmental Protection Agency (USEPA) Method TO-15 (TO-15). The TO-15 method uses a 6-liter SUMMA® passivated stainless steel canister. An evacuated SUMMA® canister (<28 inches of mercury [Hg]) will provide a recoverable whole-gas sample of approximately 5.5 liters when allowed to fill to a vacuum of 2-7 inches of Hg. The whole-air sample is then analyzed for VOCs using a quadrupole or ion-trap gas chromatograph/mass spectrometer (GS/MS) system to provide compound detection limits of 0.5 parts per billion volume (ppbv).

The following sections list the necessary equipment and detailed instructions for placing the sampling device and collecting ambient air samples for VOC analysis.

#### II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and cardiopulmonary resuscitation (CPR), as needed. ARCADIS field sampling personnel will be well versed in the relevant SOPs and possess the required skills and experience necessary to successfully complete the desired field work. ARCADIS personnel responsible for leading ambient air sample collection activities must have previous ambient air sampling experience.

#### III. Equipment List

The equipment required for ambient air sample collection is presented below:

- 6-liter, stainless steel SUMMA® canisters (order at least one extra, if feasible);
- Flow controllers with in-line particulate filters and vacuum gauges (flow controllers are pre-calibrated by the laboratory to a specified sample duration [e.g., 8-hour]). Confirm with lab that flow controller comes with in-line particulate filter and pressure gauge (order an extra set for each extra SUMMA® canister, if feasible);
- Appropriate-sized open-end wrench (typically 9/16-inch);
- Chain-of-custody (COC) form;
- Field notebook;

- Sample collection log (attached);
- Camera;
- Lock and chain; and
- Ladder or similar to hold canister above the ground surface (optional).

#### IV. Cautions

Care must be taken to minimize the potential for introducing interferences during the sampling event. As such, care must be taken to keep the canister away from public roadways to prevent collection of automobile source pollutants (unless this is the objective of the study). Care must also be taken to keep the canister away from heavy pedestrian traffic areas (e.g., main entranceways, walkways). If the canister is not to be overseen for the entire sample duration, precautions should be taken to maintain the security of the sample (e.g., do not place in areas regularly accessed by the public, fasten the sampling device to a secure object using lock and chain, label the canister to indicate it is part of a scientific project, place the canister in secure housing that does not disrupt the integrity/validity of the sampling event). Sampling personnel should not handle hazardous substances (such as gasoline), permanent marking pens, wear/apply fragrances, or smoke cigarettes/cigars before and/or during the sampling event.

Care should also be taken to ensure that the flow controller is pre-calibrated to the proper sample collection time (confirm with laboratory). Sample integrity is maintained if the sampling event is shorter than the target duration, but sample integrity can be compromised if the event is extended to the point that the canister reaches atmospheric pressure.

A Shipping Determination must be performed, by DOT-trained personnel, for all environmental and geotechnical samples that are to be shipped, as well as some types of environmental equipment/supplies that are to be shipped.

#### V. Health and Safety Considerations

Field sampling equipment must be carefully handled to minimize the potential for injury and the spread of hazardous substances.

#### VI. Procedure

#### Preparation of SUMMA®-Type Canister and Collection of Sample

- 1. Record the following information in the field notebook (contact the local airport or other suitable information source [e.g., weatherunderground.com] to obtain the following information):
  - ambient temperature;
  - barometric pressure; and
  - relative humidity.
- 2. Choose the sample location in accordance with the sampling plan. If a breathing zone sample is required, place the canister on a ladder, tripod, or other similar stand to locate the canister orifice 3 to 5 feet above ground. If the canister will not be overseen for the entire sampling period, secure the canister as appropriate (e.g., lock and chain).
- 3. Record SUMMA® canister serial number and flow controller number in the field notebook and COC form. Assign sample identification on canister ID tag and record in the field notebook, sample collection log (attached), and COC form.
- 4. Remove the brass dust cap from the SUMMA® canister. Attach the flow controller with in-line particulate filter and vacuum gauge (leave swage-lock cap on the vacuum gauge during this procedure) to the SUMMA® canister with the appropriate wrench. Tighten with fingers first, then gently with the wrench.
- 5. Open the SUMMA® canister valve to initiate sample collection. Record the date and local time (24-hour basis) of valve opening in the field notebook, sample collection log, and COC form.
- Record the initial vacuum pressure in the SUMMA® canister in the field notebook and COC form. If the initial vacuum pressure does not register less than -28 inches of Hg, then the SUMMA® canister is not appropriate for use and another canister should be used.
- 7. Take a photograph of the SUMMA® canister and surrounding area.

#### **Termination of Sample Collection**

1. Arrive at the SUMMA® canister location at least 10 to 15 minutes prior to the end of the sampling interval (e.g., 8-hour).

- Stop collecting the sample when the canister vacuum reaches approximately 2-7 inches of Hg (leaving some vacuum in the canister provides a way to verify if the canister leaks before it reaches the laboratory) or when the desired sample time has elapsed.
- 3. Record the final vacuum pressure. Stop collecting the sample by closing the SUMMA® canister valve. Record the date and local time (24-hour basis) of valve closing in the field notebook, sample collection log, and COC form.
- 4. Remove the particulate filter and flow controller from the SUMMA® canister, reinstall brass plug on canister fitting, and tighten with wrench.
- 5. Package the canister and flow controller in the shipping container supplied by the laboratory for return shipment to the laboratory. The SUMMA® canister does not require preservation with ice or refrigeration during shipment.
- 6. Complete the appropriate forms and sample labels as directed by the laboratory (e.g., affix card with string).
- 7. Complete COC forms and place requisite copies in shipping container. Close shipping container and affix custody seal to container closure. Ship to laboratory via overnight carrier (e.g., Federal Express) for analysis.

#### VII. Waste Management

No specific waste management procedures are required.

#### VIII. Data Recording and Management

Measurements will be recorded in the field notebook at the time of measurement, with notations of project name, sample date, sample start and finish times, sample location (e.g., GPS coordinates if available), canister serial number, flow controller number, initial vacuum reading, and final vacuum reading. Field sampling logs and COC records will be transmitted to the Project Manager.

#### IX. Quality Assurance

Ambient air sample analysis will be performed using USEPA Method TO-15. This method uses a quadrupole or ion-trap GC/MS with a capillary column to provide optimum detection limits. The GC/MS system requires a 1-liter gas sample (which can easily be recovered from a 6-liter canister) to provide a 0.5 ppbv detection limit. The 6-liter canister also provides several additional 1-liter samples in case subsequent re-

analyses or dilutions are required. This system also offers the advantage of the GC/MS detector, which confirms the identity of detected compounds by evaluating their mass spectra in either the SCAN or SIM mode.

#### X. References

[Click here and enter Text]



1

<b>A</b> R		Indoor/Ambient Air Sample Collection Log							
Infrastructure, environment, facilities			Sample ID:						
Client:			Outdoor/Indoor:						
Project:			Sample Intake Height:						
Location:			Miscellaneous Equipment:						
Project #:			Time On/Off:						
Samplers:			Subcontractor:						

#### Instrument Readings:

Time	Canister Pressure (inches of HG)	Temperature (F or C)	Relative Humidity (%)	Air Speed (ft/min)	Pressure Differential (inches of H20)	PID (ppm or ppb)

**SUMMA Canister Information:** 

Size (circle one): 1 L 6 L

Canister ID:

Flow Controller ID:

General Observations/Notes:

Please record current weather information including wind speed and direction, ambient temperature, barometric pressure, and relative humidity via suitable information source (e.g., weatherunderground.com).



Imagine the result

# Compositing or Homogenizing Samples

Rev. #: 01

Rev Date: March 11, 2009

SOP: Compositing or Homogenizing Samples 1 Rev. #: 01 | Rev Date: March 11, 2009

**Approval Signatures** 

Indrew Korik Prepared by:

Date: <u>3/11/09</u>

Reviewed by: Mul

Date: 3/11/09

Michael Gefell (Technical Expert)

SOP: Compositing or Homogenizing Samples 2 Rev. #: 01 | Rev Date: March 11, 2009

#### I. Scope and Application

The general procedures to be used in composting/homogenizing solid and semisolid samples are outlined below.

#### II. Personnel Qualifications

ARCADIS personnel directing, supervising, or leading compositing and/or homogenizing of samples should have a minimum of 2 years of previous field experience and current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. Field personnel will also be compliant with client-specific training requirements. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work

#### III. Equipment List

The following materials will be available, as required, when compositing or homogenizing samples.

- personal protective equipment (PPE), as specified by the site Health and Safety Plan (HASP)
- stainless steel, plastic, glass or ceramic spoon (or disposable equivalent)
- stainless steel, plastic, glass or ceramic bowl (or disposable equivalent)
- stainless steel, plastic, glass or ceramic jar/bottle (or disposable equivalent)
- shovel or trowel
- plastic sheeting
- decontamination supplies
- digital camera (if allowed by facility policy)
- appropriate sample containers and forms
- field notebook and/or personal digital assistant (PDA)

#### **IV.** Cautions

The field crew must be aware of the potential chemicals of concern (COCs), and equipped with a variety of sample homogenizing equipment. The field crew must take care not to use equipment that may react with suspected COCs. For example, stainless steel implements should not be used to homogenize strongly acidic materials.

Soil, sediment, sludge and other solid/semisolid materials that are easily mixed should be thoroughly homogenized. Excessive, vigorous mixing should be avoided as COCs can be mobilized/liberated posing a health and safety risk and diminishing the representativeness of the sample.

Implements used for compositing/homogenizing should be thoroughly decontaminated between samples. Field blanks and rinse blanks should be obtained.

A Shipping Determination must be performed, by DOT-trained personnel, for all environmental and geotechnical samples that are to be shipped, as well as some types of equipment/supplies that are to be shipped.

#### V. Health and Safety Considerations

- Sample compositing/homogenizing will be performed using procedures consistent with the project Health and Safety Plan.
- Appropriate personal protective equipment (PPE) must be worn by all field personnel within the designated work area.
- Air monitoring may be required during certain field activities as required in the Site Health and Safety Plan.

ARCADIS field personnel will be familiar and compliant with Client-specific health and safety requirements.

#### VI. Procedure

Samples may require homogenization across a given depth interval, or several discrete grabs (usually five) may be combined into a composite sample. Samples for volatile organic compound (VOC) analysis will not be homogenized or composited. The procedure for mixing samples is provided below.

SOP: Compositing or Homogenizing Samples 4 Rev. #: 01 | Rev Date: March 11, 2009

- Mix the materials in a stainless steel (or appropriate non-reactive material) bowl using a stainless steel spoon (or disposable equivalents). When dealing with large sample quantities, use disposable plastic sheeting and a shovel or trowel. Note: When preparing samples for metals analyses, do not use disposable aluminum (or metal tools or trays other than stainless steel), as it may influence the analytical results.
- 2. Flatten the pile by pressing the top without further mixing.
- 3. Divide the circular pile by into equal quarters by dividing out two diameters at right angles.
- 4. Mix each quarter individually using appropriate non-reactive bowls, spoons and/or sheeting.
- 5. Mix two quarters (as described above) to form halves, then mix the two halves to form a composite or homogenous sample.
- 6. Place composite or homogenized sample into specified containers. Remaining material will be disposed of in accordance with project requirements and applicable regulations.

#### VII. Waste Management

Investigation-derived waste will be managed as described in the Investigation-Derived Waste Handling and Storage SOP.

#### VIII. Data Recording and Management

Sample identification, interval depth (if appropriate), sample date and time will be recorded in the field notebook, the boring log, and/or the PDA. The sample will also be identified on an appropriate chain of custody form, for submittal to an analytical laboratory for analysis. Consider digital photography to record unusual field conditions or to document compliance (i.e. proper labeling and storage of drums/IDW containers).

#### IX. Quality Assurance

All materials to be re-used for sample compositing/homogenizing will be decontaminated as appropriate. Field blanks and rinse blanks will be collected to evaluate decontamination procedures and to provide an indication as to whether external contamination has potentially been introduced.

SOP: Compositing or Homogenizing Samples 5 Rev. #: 01 | Rev Date: March 11, 2009

#### X. References

Not Applicable.



Imagine the result

## Chain-of-Custody, Handling, Packing and Shipping

Rev. #: 2

Rev Date: March 6, 2009

SOP: Chain-of-Custody, Handling, Packing and Shipping 1 Rev. #: 2 | Rev Date: March 6, 2009

# Prepared by: Caron Koll Date: 3/6/09 Caron Koll Reviewed by: Date: 3/6/09 Jane Kennedy(Technical Expert)

#### Approval Signatures

SOP: Chain-of-Custody, Handling, Packing and Shipping 2 Rev. #: 2 | Rev Date: March 6, 2009

#### I. Scope and Application

This Standard Operating Procedure (SOP) describes the chain-of-custody, handling, packing, and shipping procedures for the management of samples to decrease the potential for cross-contamination, tampering, mis-identification, and breakage, and to insure that samples are maintained in a controlled environment from the time of collection until receipt by the analytical laboratory.

#### II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, Department of Transportation (DOT) training, site supervisor training, and site-specific training, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and possess the skills and experience necessary to successfully complete the desired field work.

#### III. Equipment List

The following list provides materials that may be required for each project. Project documents and sample collection requirements should be reviewed prior to initiating field operations:

- indelible ink pens (black or blue);
- polyethylene bags (resealable-type);
- clear packing tape, strapping tape, duct tape;
- chain of custody
- DOT shipping forms, as applicable
- custody seals or tape;
- appropriate sample containers and labels,;
- insulated coolers of adequate size for samples and sufficient ice to maintain 4°C during collection and transfer of samples;
- wet ice;
- cushioning and absorbent material (i.e., bubble wrap or bags);

- temperature blank
- sample return shipping papers and addresses; and
- field notebook.

#### IV. Cautions

Review project requirements and select appropriate supplies prior to field mobilization.

Insure that appropriate sample containers with applicable preservatives, coolers, and packing material have been supplied by the laboratory.

Understand the offsite transfer requirements for the facility at which samples are collected.

If overnight courier service is required schedule pick-up or know where the drop-off service center is located and the hours of operation. Prior to using air transportation, confirm air shipment is acceptable under DOT and International Air Transport Association (IATA) regulation

Schedule pick-up time for laboratory courier or know location of laboratory/service center and hours of operation.

Understand DOT and IATA shipping requirements and evaluate dangerous goods shipping regulations relative to the samples being collected (i.e. complete an ARCADIS shipping determination). Review the ARCADIS SOPs for shipping, packaging and labeling of dangerous goods. Potential samples requiring compliance with this DOT regulation include:

- Methanol preservation for Volatile Organic Compounds in soil samples
- Non-aqueous phase liquids (NAPL)

#### V. Health and Safety Considerations

Follow health and safety procedures outlined in the project/site Health and Safety Plan (HASP).

Use caution and appropriate cut resistant gloves when tightening lids to 40 mL vials. These vials can break while tightening and can lacerate hand. Amber vials (thinner glass) are more prone to breakage.

Some sample containers contain preservatives.

- The preservatives must be retained in the sample container and should in no instance be rinsed out.
- Preservatives may be corrosive and standard care should be exercised to reduce potential contact to personnel skin or clothing. Follow project safety procedures if spillage is observed.
- If sample container caps are broken discard the bottle. Do not use for sample collection.

#### VI. Procedure

#### **Chain-of-Custody Procedures**

- 1. Prior to collecting samples, complete the chain-of-custody record header information by filling in the project number, project name, and the name(s) of the sampling technician(s) and other relevant project information. Attachment 1 provides an example chain-o- custody record
- 2. Chain-of-custody information MUST be printed legibly using indelible ink (black or blue).
- 3. After sample collection, enter the individual sample information on the chain-ofcustody:
  - a. Sample Identification indicates the well number or soil location that the sample was collected from. Appropriate values for this field include well locations, grid points, or soil boring identification numbers (e.g., MW-3, X-20, SB-30). When the depth interval is included, the complete sample ID would be "SB-30 (0.5-1.0) where the depth interval is in feet. Please note it is very important that the use of hyphens in sample names and depth units (i.e., feet or inches) remain consistent for all samples entered on the chain-of-custody form. DO NOT use the apostrophe or quotes in the sample ID. Sample names may also use the abbreviations "FB," "TB," and "DUP" as prefixes or suffixes to indicate that the sample is a field blank, trip blank, or field duplicate, respectively. NOTE: The sample

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nomenclature may be dictated by the project database and require unique identification for each sample collected for the project. Consult the project data management plan for additional information regarding sample identification.

- b. List the date o sample collection. The date format to be followed should be mm/dd/yy (e.g., 03/07/09) or mm/dd/yyyy (e.g. 03/07/2009).
- c. List the time that the sample was collected. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15.
- d. The composite field should be checked if the sample is a composite over a period of time or from several different locations and mixed prior to placing in sample containers.
- e. The "Grab". field should be marked with an "X" if the sample was collected as an individual grab sample. (e.g. monitoring well sample or soil interval).
- f. Any sample preservation should be noted.
- g. The analytical parameters that the samples are being analyzed for should be written legibly on the diagonal lines. As much detail as possible should be presented to allow the analytical laboratory to properly analyze the samples. For example, polychlorinated biphenyl (PCB) analyses may be represented by entering "PCBs" or "Method 8082." Multiple methods and/or analytical parameters may be combined for each column (e.g., PCBs/VOCs/SVOCs or 8082/8260/8270). These columns should also be used to present project-specific parameter lists (e.g., Appendix IX+3 target analyte list. Each sample that requires a particular parameter analysis will be identified by placing the number of containers in the appropriate analytical parameter column. For metals in particular, indicate which metals are required.
- h. Number of containers for each method requested. This information may be included under the parameter or as a total for the sample based on the chain of custody form used.
- i. Note which samples should be used for site specific matrix spikes.
- j. Indicate any special project requirements.

- k. Indicate turnaround time required.
- I. Provide contact name and phone number in the event that problems are encountered when samples are received at the laboratory.
- m. If available attach the Laboratory Task Order or Work Authorization forms
- n. The remarks field should be used to communicate special analytical requirements to the laboratory. These requirements may be on a per sample basis such as "extract and hold sample until notified," or may be used to inform the laboratory of special reporting requirements for the entire sample delivery group (SDG). Reporting requirements that should be specified in the remarks column include: 1) turnaround time; 2) contact and address where data reports should be sent; 3) name of laboratory project manager; and 4) type of sample preservation used.
- o. The "Relinquished By" field should contain the signature of the sampling technician who relinquished custody of the samples to the shipping courier or the analytical laboratory.
- p. The "Date" field following the signature block indicates the date the samples were relinquished. The date format should be mm/dd/yyyy (e.g., 03/07/2005).
- q. The "Time" field following the signature block indicates the time that the samples were relinquished. The time value should be presented using military format. For example, 3:15 P.M. should be entered as 15:15.
- r. The "Received By" section is signed by sample courier or laboratory representative who received the samples from the sampling technician or it is signed upon laboratory receipt from the overnight courier service.
- 3. Complete as many chain-of-custody forms as necessary to properly document the collection and transfer of the samples to the analytical laboratory.
- 4. Upon completing the chain-of-custody forms, forward two copies to the analytical laboratory and retain one copy for the field records.
- 5. If electronic chain-of-custody forms are utilized, sign the form and make 1 copy for ARCADIS internal records and forward the original with the samples to the laboratory.

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#### **Handling Procedures**

- 1. After completing the sample collection procedures, record the following information in the field notebook with indelible ink:
  - project number and site name;
  - sample identification code and other sample identification information, if appropriate;
  - sampling method;
  - date;
  - name of sampler(s);
  - time;
  - location (project reference);
  - location of field duplicates and both sample identifications;
  - locations that field QC samples were collected including equipment blanks, field blanks and additional sample volume for matrix spikes; and
  - any comments.
- 2. Complete the sample label with the following information in indelible ink:
  - sample type (e.g., surface water);
  - sample identification code and other sample identification information, if applicable;
  - analysis required;
  - date;
  - time sampled; and
  - initials of sampling personnel;

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- sample matrix; and
- preservative added, if applicable.
- 3. Cover the label with clear packing tape to secure the label onto the container and to protect the label from liquid.
- 4. Confirm that all caps on the sample containers are secure and tightly closed.
- 5. In some instances it may be necessary to wrap the sample container cap with clear packing tape to prevent it from becoming loose.
- 6. For some projects individual custody seals may be required. Custody seal evidence tape may be placed on the shipping container or they may be placed on each sample container such that the cooler or cap cannot be opened without breaking the custody seal. The custody seal should be initialed and dated prior to relinquishing the samples.

#### **Packing Procedures**

Following collection, samples must be placed on wet ice to initiate cooling to 4°C immediately. Retain samples on ice until ready to pack for shipment to the laboratory.

- 1. Secure the outside and inside of the drain plug at the bottom of the cooler being used for sample transport with "Duct" tape.
- 2. Place a new large heavy duty plastic garbage bag inside each cooler
- 3. Place each sample bottle wrapped in bubble wrap inside the garbage bag. VOC vials may be grouped by sample in individual resealable plastic bags). If a cooler temperature blank is supplied by the laboratory, it should be packaged following the same procedures as the samples. If the laboratory did not include a temperature blank, do not add one. Place 1 to 2 inches of cushioning material (i.e., vermiculite) at the bottom of the cooler.
- 4. Place the sealed sample containers upright in the cooler.
- 5. Package ice in large resealable plastic bags and place inside the large garbage bag in the cooler. Samples placed on ice will be cooled to and maintained at a temperature of approximately 4°C.

SOP: Chain-of-Custody, Handling, Packing and Shipping Rev. #: 2 | Rev Date: March 6, 2009

- 6. Fill the remaining space in the cooler with cushioning material such as bubble wrap. The cooler must be securely packed and cushioned in an upright position and be surrounded (Note: to comply with 49 CFR 173.4, filled cooler must not exceed 64 pounds).
- 7. Place the completed chain-of-custody record(s) in a large resealable bag and tape the bag to the inside of the cooler lid.
- 8. Close the lid of the cooler and fasten with packing tape.
- 9. Wrap strapping tape around both ends of the cooler.
- 10. Mark the cooler on the outside with the following information: shipping address, return address, "Fragile, Handle with Care" labels on the top and on one side, and arrows indicating "This Side Up" on two adjacent sides.
- 11. Place custody seal evidence tape over front right and back left of the cooler lid, initial and date, then cover with clear plastic tape.

Note: Procedure numbers 2, 3, 5, and 6 may be modified in cases where laboratories provide customized shipping coolers. These cooler types are designed so the sample bottles and ice packs fit snugly within preformed styrofoam cushioning and insulating packing material.

#### **Shipping Procedures**

- 1. All samples will be delivered by an express carrier within 48 hours of sample collection. Alternatively, samples may be delivered directly to the laboratory or laboratory service center or a laboratory courier may be used for sample pickup.
- 2. If parameters with short holding times are required (e.g., VOCs [EnCore™ Sampler], nitrate, nitrite, ortho-phosphate and BOD), sampling personnel will take precautions to ship or deliver samples to the laboratory so that the holding times will not be exceeded.
- 3. Samples must be maintained at 4°C+2°C until shipment and through receipt at the laboratory
- 4. All shipments must be in accordance with DOT regulations and ARCADIS dangerous goods shipping SOPs.

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5. When the samples are received by the laboratory, laboratory personnel will complete the chain-of-custody by recording the date and time of receipt of samples, measuring and recording the internal temperature of the shipping container, and checking the sample identification numbers on the containers to ensure they correspond with the chain-of-custody forms.

Any deviations between the chain-of-custody and the sample containers, broken containers, or temperature excursions will be communicated to ARCADIS immediately by the laboratory.

#### VII. Waste Management

Not applicable

#### VIII. Data Recording and Management

Chain-of-custody records will be transmitted to the ARCADIS PM or designee at the end of each day unless otherwise directed by the ARCADIS PM. The sampling team leader retains copies of the chain-of-custody forms for filing in . the project file. Record retention shall be in accordance with project requirements.

#### IX. Quality Assurance

Chain-of-custody forms will be legibly completed in accordance with the applicable project documents such as Sampling and Analysis Plan (SAP), Quality Assurance Project Plan (QAPP), Work Plan, or other project guidance documents. A copy of the completed chain-of-custody form will be sent to the ARCADIS Project Manager or designee for review.

#### X. References

Not Applicable

#### SOP: Chain-of-Custody, Handling, Packing and Shipping 1 Rev. #: 2 | Rev Date: March 6, 2009

#### Attachment 1

ARCADIS	ID#:				СНА		F CUS						age	of	Lab Work	Order #
Contact & Company Name		Telephone.				Preservative Filtered (~)								Preservation Key:	Keys Container Information Key: 1. 40 ml Viai	
Address: Fox: City State Zip E-mail Address:							# of Containers Container Information								A. H <sub>2</sub> SO <sub>4</sub> B. HCL C. HNO <sub>3</sub> D. NaOH	2 1 L Amber 3 250 ml Plastic 4 500 ml Plastic
City State	Zip	E-mail Address.					PARAMETER ANALYSIS & METHOD							/	E None 5 Encore F Other 6 2 oz Glass G Other 8 2 oz Glass H Other 9, Other 10. Other Matrix Key:	
Project Name/Location (City, State) Project #												' / /	8 8 oz. Glass 9, Other			
Sample's Printed Name.		Colle		Тур	e (~)	Matrix		/	/	/	/	/			SO - Soll W - Water	SE - Sediment NL - NAPL/Oil SL - Studge SW - Sample Wipe A - Air Other:
			Time		Grab											
Special Instructions/Comments:										_] Special (	QA/QC Instru					
Laboratory Information and Receipt Lab Name: Cooler Custody Seal ( ) Printed</td <td colspan="2">Relinquished By d Name.</td> <td colspan="2">Received By Printed Name</td> <td colspan="2">Relinquished By Printed Name,</td> <td></td> <td>Laboratory Received By IName</td>				Relinquished By d Name.		Received By Printed Name		Relinquished By Printed Name,			Laboratory Received By IName					
				do sen i		Birach										
Cooler packed with ice (*)		Intact Not Intact Signab			dure:			Signature.		Signature.		Signab	urc.			
Specify Tumaround Requirements: Sample Receipt:				Fim	Firm			Firm/Couner.		Firm/Counter		Firm:				
Snipping Tracking #. Condition/Cooler Temp: Date/Temp:			Tine.			Date/Time:			Date/Time:		Date/T	ine:				

207200225 CetC AR Ferm 01.12.2007 Distribution: WHITE – Laboratory returns with results YELLOW – Lab copy PINK – Retained by BBL g/sop-library/reformatted sops 2008/general sops/1663199 - chain-of-custody, handling, packing and shipping.doc



Imagine the result

## **Field Equipment Decontamination**

Rev. #: 3

Rev Date: April 26, 2010

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**Approval Signatures** 

Date: 4/26/2010

Keith Shepherd

Reviewed by:

Prepared by:

Richard Murphy (Technical Expert)

Date: 4/26/2010

#### I. Scope and Application

Equipment decontamination is performed to ensure that sampling equipment that contacts a sample, or monitoring equipment that is brought into contact with environmental media to be sampled, is free from analytes of interest and/or constituents that would interfere with laboratory analysis for analytes of interest. Equipment must be cleaned prior to use for sampling or contact with environmental media to be sampled, and prior to shipment or storage. The effectiveness of the decontamination procedure should be verified by collecting and analyzing equipment blank samples.

The equipment cleaning procedures described herein includes pre-field, in the field, and post-field cleaning of sampling tools which will be conducted at an established equipment decontamination area (EDA) on site (as appropriate). Equipment that may require decontamination at a given site includes: soil sampling tools; groundwater, sediment, and surface-water sampling devices; water testing instruments; down-hole instruments; and other activity-specific sampling equipment. Non-disposable equipment will be cleaned before collecting each sample, between sampling events, and prior to leaving the site. Cleaning procedures for sampling equipment will be monitored by collecting equipment blank samples as specified in the applicable work plan or field sampling plan. Dedicated and/or disposable (not to be re-used) sampling equipment will not require decontamination.

#### II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training, including 40-hour HAZWOPER training, site supervisor training, and site-specific training, as needed. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and possess the skills and experience necessary to successfully complete the desired fieldwork. The project HASP and other documents will identify any other training requirements such as site specific safety training or access control requirements.

#### III. Equipment List

- health and safety equipment, as required in the site Health and Safety Plan (HASP)
- distilled water

- Non-phosphate detergent such as Alconox or, if sampling for phosphorus phosphorus-containing compounds, Luminox (or equivalent).
- tap water
- rinsate collection plastic containers
- DOT-approved waste shipping container(s), as specified in the work plan or field sampling plan (if decontamination waste is to be shipped for disposal)
- brushes
- large heavy-duty garbage bags
- spray bottles
- (Optional) Isoprophyl alcohol (free of ketones) or methanol
- Ziploc-type bags
- plastic sheeting

#### **IV.** Cautions

Rinse equipment thoroughly and allow the equipment to dry before re-use or storage to prevent introducing solvent into sample medium. If manual drying of equipment is required, use clean lint-free material to wipe the equipment dry.

Store decontaminated equipment in a clean, dry environment. Do not store near combustion engine exhausts.

If equipment is damaged to the extent that decontamination is uncertain due to cracks or dents, the equipment should not be used and should be discarded or submitted for repair prior to use for sample collection.

A proper shipping determination will be performed by a DOT-trained individual for cleaning materials shipped by ARCADIS.

#### V. Health and Safety Considerations

Review the material safety data sheets (MSDS) for the cleaning materials used in decontamination. If solvent is used during decontamination, work in a well-ventilated area and stand upwind while applying solvent to equipment. Apply solvent in a manner that minimizes potential for exposure to workers. Follow health and safety procedures outlined in the HASP.

#### VI. Procedure

A designated area will be established to clean sampling equipment in the field prior to sample collection. Equipment cleaning areas will be set up within or adjacent to the specific work area, but not at a location exposed to combustion engine exhaust. Detergent solutions will be prepared in clean containers for use in equipment decontamination.

#### **Cleaning Sampling Equipment**

- 1. Wash the equipment/pump with potable water.
- 2. Wash with detergent solution (Alconox, Liquinox or equivalent) to remove all visible particulate matter and any residual oils or grease.
- 3. If equipment is very dirty, precleaning with a brush and tap water may be necessary.
- 4. (Optional) Flush with isopropyl alcohol (free of ketones) or with methanol. This step is optional but should be considered when sampling in highly impacted media such as non-aqueous phase liquids or if equipment blanks from previous sampling events showed the potential for cross contamination of organics.
- 5. Rinse with distilled/deionized water.

#### **Decontaminating Submersible Pumps**

Submersible pumps may be used during well development, groundwater sampling, or other investigative activities. The pumps will be cleaned and flushed before and between uses. This cleaning process will consist of an external detergent solution wash and tap water rinse, a flush of detergent solution through the pump, followed

by a flush of potable water through the pump. Flushing will be accomplished by using an appropriate container filled with detergent solution and another contained filled with potable water. The pump will run long enough to effectively flush the pump housing and hose (unless new, disposable hose is used). Caution should be exercised to avoid contact with the pump casing and water in the container while the pump is running (do not use metal drums or garbage cans) to avoid electric shock. Disconnect the pump from the power source before handling. The pump and hose should be placed on or in clean polyethylene sheeting to avoid contact with the ground surface.

#### VII. Waste Management

Equipment decontamination rinsate will be managed in conjunction with all other waste produced during the field sampling effort. Waste management procedures are outlined in the work plan or Waste Management Plan (WMP).

#### VIII. Data Recording and Management

Equipment cleaning and decontamination will be noted in the field notebook. Information will include the type of equipment cleaned, the decontamination location and any deviations from this SOP. Specific factors that should be noted include solvent used (if any), and source of water.

Any unusual field conditions should be noted if there is potential to impact the efficiency of the decontamination or subsequent sample collection.

An inventory of the solvents brought on site and used and removed from the site will be maintained in the files. Records will be maintained for any solvents used in decontamination, including lot number and expiration date.

Containers with decontamination fluids will be labeled.

#### IX. Quality Assurance

Equipment blanks should be collected to verify that the decontamination procedures are effective in minimizing potential for cross contamination. The equipment blank is prepared by pouring deionized water over the clean and dry tools and collecting the deionized water into appropriate sample containers. Equipment blanks should be analyzed for the same set of parameters that are performed on the field samples collected with the equipment that was cleaned. Equipment blanks are collected per equipment set, which represents all of the tools needed to collect a specific sample.

#### X. References

- USEPA Region 9, Field Sampling Guidance #1230, Sampling Equipment Decontamination.
- USEPA Region 1, Low Stress (low flow) Purging and Sampling Procedure for the Collection of Groundwater Samples from Monitoring Wells.



Imagine the result

## Investigation-Derived Waste Handling and Storage

Rev. #: 2

Rev Date: March 6, 2009

SOP: Investigation-Derived Waste Handling and Storage 1 Rev. #: 2 | Rev Date: March 6, 2009

**Approval Signatures** 

Johnew Kanik Date: \_\_\_\_\_ Prepared by: <u>3/6/09</u> Reviewed by: Date: 3/6/09 (Technical Expert)

#### I. Scope and Application

The objective of this Standard Operating Procedure (SOP) is to describe the procedures to manage investigation-derived wastes (IDW), both hazardous and nonhazardous, generated during site activities, which may include, but are not limited to drilling, trenching/excavation, construction, demolition, monitoring well sampling, soil sampling, decontamination and remediation. Please note that this SOP is intended for materials that have been deemed a solid waste as defined by 40 CFR § 261.2 (which may includes liquids, solids, and sludges). In some cases, field determinations will be made based on field screening or previous data that materials are not considered a solid waste. IDW may include soil, groundwater, drilling fluids, decontamination liquids, personal protective equipment (PPE), sorbent materials, construction and demolition debris, and disposable sampling materials that may have come in contact with potentially impacted materials. IDW will be collected and staged at the point of generation. Quantities small enough to be containerized in 55-gallon drums will be taken to a designated temporary storage area (discussed in further detail under Drum Storage) onsite pending characterization and disposal. Waste materials will be analyzed for constituents of concern to evaluate proper disposal methods. PPE and disposable sampling equipment will be placed in DOT-approved drums prior to disposal and typically does not require laboratory analysis. This SOP describes the necessary equipment, field procedures, materials, regulatory references, and documentation procedures necessary for proper handling and storage of IDW up to the time it is properly disposed. The procedures for handling IDW are based on the United States Environmental Protection Agency's Guide to Management of Investigation Derived Wastes (USEPA, 1992). IDW is assumed to be contaminated with the site constituents of concern (COCs) until analytical evidence indicates otherwise. IDW will be managed to ensure the protection of human health and the environment and will comply with all applicable or relevant and appropriate requirements (ARAR). The following Laws and Regulations on Hazardous Waste Management are potential ARAR for this site.

#### State Laws and Regulations

 To Be Determined Based on Location of Site and Location of Treatment, Storage, and/or Disposal Facility (TSDF) to be utilized

#### **Federal Laws and Regulations**

- Resource Conservation and Recovery Act (RCRA) 42 USC § 6901-6987
- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA) 42 USC § 9601-9675

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- Superfund Amendments and Reauthorization Act (SARA)
- Department of Transportation (DOT) Hazardous Materials Transportation

Pending characterization, IDW will be stored appropriately within each area of contamination (AOC). Under RCRA, "storage" is defined as the holding of hazardous waste for a temporary period, at the end of which the hazardous waste is treated, disposed of, or stored elsewhere" (40 CFR § 260.10). The onsite waste staging area will be in a secure and controlled area. Waste characterization can either be based on generator knowledge, such as using materials safety data sheets (MSDS'), or can be based upon analytical results. The laboratory used for waste characterization analysis must have the appropriate state and federal certifications and be approved by ARCADIS and Client. IDW will be classified as RCRA hazardous or non-regulated under RCRA based on the waste characterization.

If IDW is characterized as RCRA hazardous waste, RCRA and DOT requirements must be followed for packaging, labeling, transporting, storing, and record keeping as described in 40 CFR § 262 and 49 CFR § 171-178. Wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved packaging. Waste material classified as RCRA non-hazardous may be handled and disposed of as an industrial waste.

Liquid wastes judged to potentially meet the criteria for hazardous wastes shall be stored in DOT approved 55 gallon drums or other approved containers that are compatible with the type of material stored therein. Solid materials deemed to potentially meet hazardous criteria will be drummed where practicable. Large quantities of potentially hazardous solid materials must be containerized (such as in a roll-off box) for up to a maximum of 90 or 180 days as described in the Excavated Solids Section. Waste material classified as non-hazardous may be handled and disposed of as an industrial waste and is not subject to the 90-day or 180-day on-site storage limitation.

This is a standard (i.e., typically applicable) operating procedure which may be varied or changed as required, dependent upon site conditions, equipment limitations, or limitations imposed by the procedure. The ultimate procedure employed will be documented in the project work plans or reports. If changes to the sampling procedures are required due to unanticipated field conditions, the changes will be discussed with the Project Manager and Client as soon as practicable and documented in the report.

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### II. Personnel Qualifications

ARCADIS field sampling personnel will have current health and safety training including 40-hour HAZWOPER training, site supervisor training, site-specific training, first aid, and CPR, as needed. ARCADIS personnel may sign manifests on a case-to-case basis for clients, provided the appropriate agreement is in place between ARCADIS and the client documenting that ARCADIS is not the generator, but is acting as authorized representative for the generator. ARCADIS personnel who sign hazardous waste manifests will have the current DOT hazardous materials transportation training according to 49 CFR § 172.704. ARCADIS field personnel will also comply with client-specific training such as LPS. In addition, ARCADIS field sampling personnel will be versed in the relevant SOPs and posses the required skills and experience necessary to successfully complete the desired field work.

### III. Equipment List

The following materials, as required, shall be available for IDW handling and storage:

Appropriate personal protective equipment as specified in the Site Health and Safety Plan

- 55-gallon steel drums, DOT 1A2 or equivalent
- ¾ -inch socket wrench
- Hammer
- Leather gloves
- Drum dolly
- Appropriate drum labels (outdoor waterproof self adhesive)
- Polyethylene storage tank
- Appropriate labeling, packing, chain-of-custody forms, and shipping materials as specified in the *Chain-of-Custody* SOP and *Field Sampling Handling, Packing, and Shipping* SOP.
- Indelible ink and/or permanent marking pens
- Plastic sheeting

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- Appropriate sample containers, labels, and forms
- Stainless-steel bucket auger
- Stainless steel spatula or knife
- Stainless steel hand spade
- Stainless steel scoop
- Digital camera
- Field logbook.

## IV. Cautions

- Filled drums can be very heavy, always use appropriate moving techniques and equipment.
- Similar media will be stored in the same drums to aid in sample analysis and disposal.
- Drum lids must be secured to prevent rainwater from entering the drums.
- Drums containing solid material may not contain any free liquids.
- Waste containers stored for extended periods of time may be subject to deterioration. Drum over packs may be used as secondary containment.
- All drums must be in good condition to prevent potential leakage and facilitate subsequent disposal. Inspect the drums for dents and rust, and verify the drum has a secure lid prior to use.

#### V. Health and Safety Considerations

- Appropriate personal protective equipment must be worn by all field personnel within the designated work area.
- Air monitoring may be required during certain field activities as required in the Site Health and Safety Plan.

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- If excavating in potentially hazardous areas is possible, contingency plans should be developed to address the potential for encountering gross contamination or non-aqueous phase liquids.
- ARCADIS field personnel will be familiar and compliant with Client-specific health and safety requirements such as Chevron's hand safety policy including the prohibition of fixed and/or folding blade knives.

## VI. Procedure

Waste storage and handling procedures to be used depend upon the type of generated waste. For this reason, IDW should be stored in a secure location onsite in separate 55-gallon storage drums, solids can be stockpiled onsite (if non-hazardous), and purge water may be stored in polyethylene tanks. Waste materials such as broken sample bottles or equipment containers and wrappings will be stored in 55-gallon drums unless they were not in contact with sample media.

#### Management of IDW

Minimization of IDW should be considered by the Project Manager during all phases of the project. Site managers may want to consider techniques such as replacing solventbased cleaners with aqueous-based cleaners for decontamination of equipment, reuse of equipment (where it can be decontaminated), limitation of traffic between exclusion and support zones, and drilling methods and sampling techniques that generate little waste. Alternative drilling and subsurface sampling methods may include the use of small diameter boreholes, as well as borehole testing methods such as a core penetrometer or direct push technique instead of coring (EPA, 1993).

#### **Drum Storage**

Drums containing hazardous waste shall be stored in accordance with the requirements of 40 CFR 265 Subpart I (for containers) and 265 Subpart DD (for containment buildings). All 55-gallon drums will be stored at a secure, centralized onsite location that is readily accessible for vehicular pick-up. Drums confirmed as, or believed to contain hazardous waste will be stored over an impervious surface provided with secondary containment. The storage location will, for drums containing liquid, have a containment system that can contain at least the larger of 10% of the aggregate volume of staged materials or 100% of the volume of the largest container. Drums will be closed during storage and be in good condition in accordance with the Guide to Management of Investigation-Derived Wastes (USEPA, 1992).

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#### **Hazardous Waste Determination**

Waste material must be characterized to determine if it meets any of the federal definitions of hazardous waste as required by 40 CFR § 262.11. If the waste does not meet any of the federal definitions, it must then be established if any state-specific hazardous waste criteria exist/apply.

#### **Generator Status**

Once hazardous waste determination has been made, the generator status will be determined. Large quantity generators (LQG) are generators who generate more than 1,000 kilograms of hazardous waste in a calendar month. Small quantity generators (SQG) of hazardous waste are generators who generate greater than 100 kilograms but less than 1,000 kilograms of hazardous waste in a calendar month. Conditionally exempt small quantity generators (CESQG) are generators who generate less than 100 kilograms of hazardous waste per month. Please note that a generator status may change from month to month and that a notice of this change is usually required by the generator's state agency.

### **Accumulation Time for Hazardous Waste**

A LQG may accumulate hazardous waste on site for 90 days or less without a permit and without having interim status provided that such accumulation is in compliance with specifications in 40 CFR § 262.34. A SQG may accumulate hazardous waste on site for 180 days or less without a permit or without having interim status subject to the requirements of 40 CFR § 262.34(d). CESQG requirements are found in 40 CFR § 261.5. **NOTE**: The CESQG and SQG provisions of 40 CFR § 261.5, 262.20(e), 262.42(b) and 262.44 may not be recognized by some states (e.g. Rhode Island). **State-specific regulations must be reviewed and understood prior to the generation of hazardous waste.** 

#### Satellite Accumulation of Hazardous Waste

Satellite accumulation (SAA) shall mean the accumulation of as much as fifty-five (55) gallons of hazardous waste, or the accumulation of as much as one quart of acutely hazardous waste, in containers at or near any point of generation where the waste initially accumulates, which is under the control of the operator of the process generating the waste, without a permit or interim status and without complying with the requirements of 40 CFR § 262.34(a) and without any storage time limit, provided that the generator complies with 40 CFR § 262.34(c)(1)(i).

Once more than 55 gallons of hazardous waste accumulates in SAA, the generator has three days to move this waste into storage.

Storage recommendations for hazardous waste include:

- Ignitable Hazardous wastes must be >50 feet from the property line per 40 CFR § 265.176 (LQG generators only).
- Hazardous waste must be stored on a concrete slab (asphalt is acceptable if there are no free liquids in the waste) per 40 CFR § 265.176.
- Drainage must be directed away from the accumulation area.
- Area must be properly vented.
- Area must be secure.

### **Drum/Container Labeling**

Drums will be labeled on both the side and lid of the drum using a permanent marking pen. Old drum labels must be removed to the extent possible, descriptions crossed out should any information remain, and new labels affixed on top of the old labels. Other containers used to store various types of waste (polyethylene tanks, roll-off boxes, end-dump trailers, etc.) will be labeled with an appropriate "Waste Container" or "Testing in Progress" label pending characterization. Drums and containers will be labeled as follows:

- Appropriate waste characterization label (Testing In Progress, Hazardous, or Non-Hazardous)
- Waste generator's name (e.g., client name)
- Project name
- Name and telephone number of ARCADIS project manager
- Composition of contents (e.g., used oil, acetone 40%, toluene 60%)
- Media (e.g., solid, liquid)
- Accumulation start date

 Drum number of total drums as reconciled with the Drum Inventory maintained in the field log book.

IDW containers will remain closed except when adding or removing waste. Immediately upon beginning to place waste into the drum/container, a "Waste Container" or "Testing in Progress" label will be filled out to include the information specified above, and affixed to the container. Once the contents of the container are identified as either non-hazardous or hazardous, the following additional labels will be applied. Containers with waste determined to be non-hazardous will be labeled with a green and white "Non-Hazardous Waste" label over the "Waste Container" label. Containers with waste determined to be hazardous will be stored in an onsite storage area and will be labeled with the "Hazardous Waste" label and affixed over the "Waste Container" label. The ACCUMULATION DATE for the hazardous waste is the date the waste is first placed in the container and is the same date as the date on the "Waste Container" label. DOT hazardous class labels must be applied to all hazardous waste containers for shipment offsite to an approved disposal or recycling facility. In addition a DOT proper shipping name shall be included on the hazardous waste label. The transporter should be equipped with the appropriate DOT placards. However, placarding or offering placards to the initial transporter is the responsibility of the generator per 40 CFR § 262.33.

#### **Inspections and Documentation**

All IDW will be documented as generated on a Drum Inventory Log maintained in the field log book. The Drum Inventory will record the generation date, type, quantity, matrix and origin (e.g. Boring-1, Test Pit 3, etc) of materials in every drum, as well as a unique identification number for each drum. The drum inventory will be used during drum pickup to assist with labeling of drums. The drum storage area and any other areas of temporarily staged waste, such as soil/debris piles, will be inspected weekly. The weekly inspections will be recorded in the field notebook or on a Weekly Inspection Log. Digital photographs will be taken upon the initial generation and drumming/staging of waste, and final labeling after characterization to document compliance with labeling and storage protocols, and condition of the container. Evidence of damage, tampering or other discrepancy should be documented photographically.

#### **Emergency Response and Notifications**

Specific procedures for responding to site emergencies will be detailed in the HASP. If the generator is designated as a LQG, a Contingency Plan will need to be prepared to include emergency response and notification procedures per 40 CFR § 265 Subpart D. In the event of a fire, explosion, or other release which could threaten human health

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outside of the site or when Client or ARCADIS has knowledge of a spill that has reached surface water, Client or ARCADIS must immediately notify the National Response Center (800-424-8802) in accordance with 40 CFR § 262.34. Other notifications to state agencies may also be necessary.

#### **Drilling Soil Cuttings and Muds**

Soil cuttings are solid to semi-solid soils generated during trenching activities, subsurface soil sampling, or installation of monitoring wells. Depending on the drilling method, drilling fluids known as "muds" may be used to remove soil cuttings. Drilling fluids flushed from the borehole must be directed into a settling section of a mud pit. This allows reuse of the decanted fluids after removal of the settled sediments. Soil cuttings will be labeled and stored in 55-gallon drums with bolt-sealed lids.

#### **Excavated Solids**

Excavated solids may include, but are not limited to soil, fill and construction and demolition debris. Excavated solids may be temporarily stockpiled onsite as long as the material is a RCRA non-hazardous waste and the solids will be treated onsite pursuant to a certified, authorized, or permitted treatment method, or properly disposed off-site. Stockpiled materials characterized as hazardous must be immediately containerized and removed from the site within 90 days of generation (except for soils using satellite accumulation). Excavated solids should be stockpiled and maintained in a secure area onsite. At a minimum, the floor of the stockpile area will be covered with a 20-mil high density polyethylene liner that is supported by a foundation or at least a 60-mil high density polyethylene liner that is not supported by a foundation. The excavated material will not contain free liquids. The owner/operator will provide controls for windblown dispersion, run-on control, and precipitation runoff. The run-on control system will prevent flow onto the active portion of the pile during peak discharge from at least a 25-year storm and the run-off management system will collect and control at least the water volume resulting from a 24-hour, 25-year storm (EPA, 1992). Additionally, the stockpile area will be inspected on a weekly basis and after storm events. Individual states may require that the stockpile be inspected/certified by a licensed professional engineer. Stockpiled material will be covered with a 6-mil polyvinyl chloride (PVC) liner. The stockpile cover will be secured in place with appropriate material (concrete blocks, weights, etc.) to prevent the movement of the cover. Excavated solids may also be placed in roll off containers and covered with a 6-mil PVC liner pending results for waste characterization.

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#### **Decontamination Solutions**

Decontamination solutions are generated during the decontamination of personal protective equipment and sampling equipment. Decontamination solutions may range from detergents, organic solvents and acids used to decontaminate small field sampling equipment to steam cleaning rinsate used to wash heavy field equipment. These solutions are to be labeled and stored in 55-gallon drums with bolt-sealed lids.

#### **Disposable Equipment**

Disposable equipment includes personal protective equipment (tyvek coveralls, gloves, booties and APR cartridges) and disposable sampling equipment such as trowels or disposable bailers. If the media sampled exhibits hazardous characteristics per results of waste characterization sampling, disposable equipment will also be disposed of as a hazardous waste. These materials will be stored onsite in labeled 55-gallon drums pending analytical results for waste characterization.

#### **Purge Water**

Purge water includes groundwater generated during well development, groundwater sampling, or aquifer testing. The volume of groundwater generated will dictate the appropriate storage procedure. Monitoring well development and groundwater sampling may generate three well volumes of groundwater or more. This volume will be stored in labeled 55-gallon drums. Aquifer tests may generate significantly greater volumes of groundwater depending on the well yield and the duration of the test. Therefore, large-volume portable polyethylene tanks will be considered for temporary storage pending groundwater-waste characterization.

### Purged Water Storage Tank Decontamination and Removal

The following procedures will be used for inspection, cleaning, and offsite removal of storage tanks used for temporary storage of purge water. These procedures are intended to be used for rented portable tanks such as Baker Tanks or Rain for Rent containers. Storage tanks will be made of inert polyethylene materials.

The major steps for preparing a rented tank for return to a vendor include characterizing the purge water, disposing of the purge water, decontaminating the tank, final tank inspection, and mobilization. Decontamination and inspection procedures are describe in further detail below.

 Tank Cleaning: Most vendors require that tanks be free of any sediment and water before returning, a professional cleaning service may be required. Each

specific vendor should be consulted concerning specific requirements for returning tanks.

 Tank Inspection: After emptying the tank, purged water storage tanks should be inspected for debris, chemical staining, and physical damage. The vendors require that tanks be returned in the original condition (i.e., free of sediment, staining and no physical damage).

### VII. Waste Characterization Sampling and Shipping

### Soil/Solids Characterization

Waste characterization will be conducted in accordance with waste hauler, waste handling facility, and state/federal requirements. In general, RCRA hazardous wastes are those solid wastes determined by a Toxicity Characteristic Leaching Procedure (TCLP) test or to contain levels of certain toxic metals, pesticides, or other organic chemicals above specific federally regulated thresholds. If the one or more of 40 toxic compounds listed in Table I of 40 CFR § 261.24 are detected in the sample at levels above the maximum unregulated concentrations, the waste must be characterized as a toxic hazardous waste. Wastes can also be considered "listed" hazardous waste depending on site-specific processes.

Composite soil samples will be collected at a frequency of one sample per 10 cubic yard basis for stockpiled soil or one per 55-gallon drum for containerized. A four point composite sample will be collected per 10 cubic yards of stockpiled material and for each drum. Sample and composite frequencies may be adjusted in accordance with the waste handling facility's requirements. Waste characterization samples may be analyzed for the TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

#### Wastewater Characterization

Waste characterization will be conducted in accordance with the requirements of the waste hauler, waste handling facility, and state/federal governments. In general, purge water should be analyzed by methods appropriate for the known contaminants, if any, that have been historically detected in the monitoring wells. Samples will be collected and analyzed in accordance with the requirements of the waste disposal facility.

Wastewater characterization samples may be analyzed for TCLP volatile organic compounds (VOCs), TCLP semi-volatile organic compounds (SVOCs), TCLP RCRA

metals, and polychlorinated biphenyls, as well as corrosivity (pH), reactivity and flammability (flashpoint). Additional samples may be collected and analyzed by the laboratory on a contingency basis.

### Sample Handling and Shipping

All samples will be appropriately labeled, packed, and shipped, and the chain-ofcustody will be filled out in accordance with the Chain-of-Custody SOP and Field Sampling Handling, Packing, and Shipping SOP and Hazardous Materials Packaging and Shipping SOP.

It should be noted that additional training is required for packaging and shipping of hazardous and/or dangerous materials. Please reference the following ARCADIS intranet team page for more information: http://team/sites/hazmat/default.aspx.

### Preparing Waste Shipment Documentation (Hazardous and Non-Hazardous)

Waste profiles will be prepared by the ARCADIS PM and forwarded, along with laboratory analytical data to the Client PM for approval/signature. The Client PM will then return the profile to ARCADIS who will then forward to the waste removal contractor for preparation of a manifest. The manifest will be reviewed by ARCADIS prior to forwarding to the Client PM for approval. Upon approval of the manifest, the Client PM will return the original signed manifest directly to the waste contractor or to the ARCADIS PM for forwarding to the waste contractor.

Final drum labeling and pickup will be supervised by an ARCADIS representative who is experienced with waste labeling procedures. The ARCADIS representative will have a copy of the drum inventory maintained in the field book and will reconcile the drum inventory with the profile numbers on the labels and on the manifest. Different profile numbers will be generated for different matrices or materials in the drums. For example, the profile number for drill cuttings will be different than the profile number for purge water. When there are multiple profiles it is critical that the proper label, with the profile number appropriate to a specific material be affixed to the proper drums. A copy of the ARCADIS drum inventory will be provided to the waste transporter during drum pickup and to the facility receiving the waste.

## VIII. Data Recording and Management

Waste characterization sample handling, packing, and shipping procedures will be documented in accordance with the *Quality Assurance Project Plan*, if one exists. Copies of the chains-of-custody forms will be maintained in the project file.

Following waste characterization, IDW containers will be re-labeled with the appropriate waste hazardous or non-hazardous waste labels and the client will initiate disposal at the appropriate waste disposal facility.

## IX. Quality Assurance

The chain-of-custody and sample labels for waste characterization samples will be filled out in accordance with the *Quality Assurance Project Plan*.

### X. References

United States Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes. Office of Remedial and Emergency Response. Hazardous Site Control Division. January 1992.

USEPA. 1991. *Guide to Discharging CERCLA Aqueous Wastes to Publicly Owned Treatment Works (POTWs)*. Office of Remedial and Emergency Response. Hazardous Site Control Division 0S-220W. March 1991.

# Attachment B-2

National Grid – Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites

# Field Descriptions of Samples for Former Manufactured Gas Plant (MGP) Sites

# SOIL SAMPLE DESCRIPTIONS

It is important that descriptive qualifiers are consistently used to characterize degree and nature of contaminant impacts and visual-manual soil classification. The following presents some examples of descriptive qualifiers.

# SOIL LOGGING

- All soils are to be logged using the **Unified Soil Classification** (ASTM D 2488 field descriptions)
- PID or FID used to screen all soil samples (Jar Headspace method) maximum readings should be recorded and included on the logs. PID/FID to be calibrated daily at a minimum
- Moisture terms are: Dry, Moist, and Wet
- **Color terms** use geotechnical color charts colors may be combined: e.g. redbrown. Color terms should be used to describe the "natural color" of the sample as opposed to staining caused by contamination (see below)
- Log of each sample interval should be prepared as follows:

[Coarse Grained Example] NARROWLY GRADED SAND (SP); mostly fine sand; <5% fines; red-brown, moist, environmental/depositional/geologic descriptions.

[Fine Grained Example] SANDY SILT (ML); heterogeneous till structure, nonplastic, ~30% fine to coarse, subangular sand; ~10% subangular fine gravel, max. size ~ 10 mm; brown; environmental/depositional/geologic descriptions.

- **Representativeness** Soil logs should include particular notes if the field representative believes that there is a possibility the soil sample being described is not representative of the interval sampled.
- Intervals for Description if using a 2' (split spoon) or 4' (Macro-core) long sampler – the field description should not necessarily be for the entire sample interval. It is important to look for, identify, and describe small-scale units and changes within each sample interval.

# **DESCRIPTION OF CONTAMINANTS**

# **Visible Contamination Descriptors**

- **Sheen** iridescent petroleum-like sheen. Not to be used to describe a "bacterial sheen", which can be distinguished by its tendency to break up on the water surface at angles whereas petroleum sheen will be continuous and will not break up. A field test for sheen is to put a soil sample in a jar of water and shake the sample (jar shake test), then observe the presence/absence of sheen on the surface of the water in the jar.
- **Stained** used w/ color (i.e. black or brown stained) to indicate that the soil matrix is stained a color other than the natural (unimpacted) color of the soil.
- **Coated** soil grains are coated with tar/free product there is not sufficient freephase material present to saturate the pore spaces.
- **Blebs** observed discrete sphericals of tar/free product but for the most part the soil matrix was not visibly contaminated or saturated. Typically this is residual product.
- **Saturated** the entirety of the pore space for a sample is saturated with the tar/free product. Care should be taken to ensure that you're not observing water saturating the pore spaces if you use this term. Depending on viscosity, tar/free-phase saturated materials may freely drain from a soil sample.
- **Oil**. Used to characterize free and/or residual product that exhibits a distinct fuel oil or diesel fuel like odor; distinctly different from MGP-related odors/impacts.
- **Tar**. Used to describe free and/or residual product that exhibits a distinct "coal tar" type odor (e.g. naphthalene-like odor). Colors of product can be brown, black, reddish-brown, or gold.
- **Solid Tar**. Used to describe product that is solid or semi-solid phase. The magnitude of the observed solid tar should be described (e.g. discrete granules or a solid layer).
- **Purifier Material**. Purifier material is commonly brown/rust or blue/green wood chips or granular material. It is typically associated with a distinctive sulfur-like odor. Other colors may be present.

# **Olfactory Descriptors**

- Use terms such as " tar-like odor" or "naphthalene-like odor" or "fuel oil-like odor" that provide a qualitative description (opinion) as to the possible source of the odor.
- Use modifiers such as strong, moderate, faint to indicate intensity of the observed odor.

# DNAPL/LNAPL

• A jar shake test should be performed to identify and determine whether observed tar/free-phase product is either denser or lighter than water. In addition, MGP residues can include both light and dense phases - this test can help determine if both light and dense phase materials are present at a particular location.

**Viscosity of Free-Phase Product** – If free-phase product/tar is present a qualitative description of viscosity should be made. Descriptors such as:

- Highly viscous (e.g. taffy-like)
- Viscous (e.g. No. 6 fuel oil or bunker crude like)
- Low viscosity (e.g. No. 2 fuel oil like)

# **GROUNDWATER SAMPLING OBSERVATIONS**

• Any observations of sheen, blebs, free-phase product/tar, staining or coating of the sampling equipment, odor, etc. that are made during sampling of groundwater are to be included in the groundwater sample collection log.

# Appendix C

Quality Assurance Project Plan



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# Appendix C Quality Assurance Project Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

September 1, 2011

Christopher D. Keen Senior Scientist

, Jeldman

Steven M. Feldman Principal Scientist

#### Appendix C Quality Assurance Project Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

Prepared for: National Grid

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- Table C-4DPCB Analytical QC Limits, Quality Assurance Project Plan (QAPP),<br/>RI Work Plan, Former Dangman Park Manufactured Gas Plant Site,<br/>Brooklyn, New York
- Table C-4EMetals and Cyanide Analytical QC Limits, Quality Assurance Project<br/>Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured<br/>Gas Plant Site, Brooklyn, New York

#### Attachments

- C-1 Project Organizational Chart
- C-2 Field Forms
- C-3 Chain-of-Custody Form
- C-4 TestAmerica Laboratories, Inc. Quality Assurance Manual and Standard Operating Procedures (SOPs)
- C-5 Alpha Analytical, Inc. Quality Systems Manual and SOP

# Appendix C Quality Assurance Project Plan

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## 1. Introduction

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this Quality Assurance Project Plan (QAPP) as a component of the Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York. The QAPP presents the quality assurance/quality control (QA/QC) procedures to be used during implementation of the RI Work Plan.

The overall QAPP objective is to ensure that data produced as a result of the various sampling and monitoring, including soil, groundwater, soil vapor, and ambient (outdoor) air is of the highest quality and usable for the intended purpose. For the purposes of this QAPP, soil vapor and ambient air samples will be collectively referred to as air samples. This QAPP has been prepared in accordance with the United States Environmental Protection Agency (USEPA) guidance entitled Guidance for Quality Assurance Project Plans EPA QA/G-5 (USEPA, 2002), the New York State Department of Environmental Conservation (NYSDEC) DER-10 Technical Guidance for Site Investigation and Remediation (NYSDEC, 2010), and considering requirements of the Multi-Site Order on Consent (ACO) and Administrative Settlement (Index # A2-0552-0606). This QAPP presents project organization and responsibilities, and QA/QC protocols related to field sampling and analysis activities associated with various sampling and monitoring requirements. The procedures in this QAPP will be implemented to ensure that precision, accuracy, representativeness, completeness, and comparability (PARCC parameters) of the data are documented, as applicable, and that data meet project requirements.

The QAPP will be used in conjunction with the RI Work Plan, the Field Sampling Plan (FSP), the Community Air Monitoring Plan (CAMP), and the Health and Safety Plan (HASP). The RI Work Plan presents the Site background and defines the field sampling program. The FSP describes the methods and procedures to be used for environmental sample collection during implementation of the RI field activities. The CAMP provides procedures to protect the downwind communities from potential airborne releases of constituents of concern during RI activities. The FSP, CAMP, and HASP are provided as Appendices B, D, and E of the RI Work Plan, respectively.

## 2. Site Description

The Site is located at 486 Neptune Avenue in the Borough of Brooklyn, New York City, Kings County, New York and occupies portions of two parcels that are identified by Tax Map Number: Block 7273, Lots 1 and 25. As shown on Figure 1 of the RI Work Plan,

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the Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is generally flat with an elevation of approximately 9 feet above mean sea level (msl). The closest natural surface water body is Coney Island Creek, which is located approximately 0.25 miles to the northwest of the Site.

The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5<sup>th</sup> Street to the east, a residential parcel to the south, and a commercial parcel to the west. Currently, the Site is developed with a shopping center and a parking lot for a high-rise apartment building.

## 3. Remedial Investigation Activities

The primary objectives of the proposed RI activities are listed in Section 1.1 of the RI Work Plan. Sample collection efforts include obtaining discrete soil samples from soil borings, groundwater samples from temporary and permanent monitoring wells, air samples (soil vapor samples from temporary soil vapor points, ambient air quality samples), and investigation-derived waste (IDW) solid and liquid media samples for waste characterization purposes.

Samples collected during the investigation will be analyzed in accordance with USEPA SW-846 Test Methods for Evaluating Solid Waste and USEPA Compendium Method TO-15, with New York State Department of Environmental Conservation (NYSDEC) Analytical Services Protocol (ASP) Revision 2005 (or most recent version).

## 4. Project Organization and Responsibilities

A Project Organizational Chart is provided as Attachment C-1. The responsibilities of the key project personnel are detailed below.

 The Project Manager is responsible for the following: overseeing the implementation of the project tasks, overall project coordination, adherence to the project schedules, directing, reviewing, and assessing the adequacy of the performance of the Task Managers assigned to the project, implementing corrective action (if warranted), reviewing reports, and maintaining full and orderly project documentation. The Project Manager will review all documents and other correspondence concerning the activities performed pursuant to the project (i.e., all activities associated with the Site). The Project Manager is also

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responsible for the overall QA including technical adequacy of the project activities and reports and conformance to the scope of work.

- The Task Manager(s) is responsible for the following: field activity QA/QC, task coordination, adherence to the project schedules, directing, reviewing, and assessing the adequacy of the performance of the technical staff and subcontractors assigned to the project (if warranted), interacting with the Project Manager, preparing reports, and maintaining full and orderly project documentation.
- The project team members include the task managers, field hydrogeologists, sampling team/field technicians, engineers, risk assessors, and support staff (e.g., data processors, project assistants, and in-house experts in engineering, etc.) who are qualified to oversee/perform the work, as appropriate, and will be responsible for work in their respective specialty areas. Project team members will be on-site to supervise all activities specified in the RI Work Plan.
- The Project QA Officer is responsible for performing systems auditing, interfacing with the analytical laboratory to make requests and resolve problems, interfacing with the data validator, and developing a project-specific data usability summary report (DUSR).
- The Site Health and Safety Officer is responsible for implementing the sitespecific health and safety directives in the Health and Safety Plan (HASP – see RI Work Plan Appendix E) and for contingency response.
- The Data Validator is responsible for review of laboratory data for compliance with the QA objectives for analytical performance and the PARCC parameters (i.e., precision, accuracy, representativeness, completeness, and comparability) as set forth in this QAPP, and notifications to the Project QA Officer and Project Manager of any QC deficiencies that impact data usability.

## 5. Quality Objectives and Criteria for Measurement Data

The overall QA objective for this aspect of the project is to select and implement procedures for field measurements, sampling, and analytical testing that will provide data of known quality to support the intended use of the information.

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The data quality objective (DQO) process, as described in the USEPA guidance entitled <u>Guidance on Systematic Planning Using the Data Quality Objectives Process</u> <u>EPA QA/G-4</u> (USEPA, 2006), is intended to provide a "logical framework" for planning field investigations. The following section addresses, in turn, each of the seven sequential steps in the EPA QA/G-4 DQO process.

## Step 1: State the Problem

The RI will be conducted at the Site to determine the nature and extent of MGP-related constituents of concern (COCs) in on-site soil, groundwater, and soil vapor. The sampling and analysis program is intended to generate data for the Site database that may potentially support further investigation work, if necessary.

## Step 2: Identify the Goal of the Study

The goal of the study is to determine the nature and extent of MGP-related COCs in on-site soil, groundwater, and soil vapor and to assess potential impacts to human health and the environment as a result of the release of COCs at the Site. The initial use of the data is descriptive (distribution and concentration). Subsequent to a review of the descriptive information, an evaluation will be performed based on the findings of the RI.

## Step 3: Identify Information Inputs

Decision inputs incorporate both concentration and distribution. A fundamental basis for decision-making is that a sufficient number of data points of acceptable quality are available from the investigation to support the decision. Thus, the necessary inputs for the decision are: 1) the proportion of non-rejected (usable) data points; and 2) the quantity of data needed to thoroughly determine the nature and extent of MGP-related COCs at the Site.

The soil, groundwater, and soil vapor sample data will be compared to the applicable New York State standards, criteria, and guidance (SCGs). The data will be evaluated for completeness, general conformance with requirements of this QAPP, and consistency among data sets as appropriate.

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### Step 4: Define the Boundaries of the Study

The Site is predominantly covered by paved surfaces and buildings. The limits of the Site were approximated from a review of historical Sanborn fire insurance maps and recent aerial photographs. The study boundaries will include subsurface soils, groundwater, and air within the on-site area.

### Step 5: Develop the Analytic Approach

The decision on whether data can be used in the Site evaluation will be based on the validation results. Following validation, the data will be flagged, as appropriate, and any use restrictions noted. The sampling plan has been devised so that the loss of any single data point will not hinder description of the distribution of COCs or the evaluation of further investigation activity. Given this, a reasonable decision rule would be that 90% of the data points not be rejected and deemed unusable for evaluation purposes. Applicable actions would be evaluated, if needed, based on the results of the RI.

SCGs have been identified for the Site that pertain to meeting applicable regulations and RI objectives. The data will be compared to the applicable SCGs to evaluate COCs that exceed their respective SCG. The SCGs for the Site soils are based upon the selection of applicable values from the New York State Codes, Rules and Regulations Title 6 (6 NYCRR) Part 375 Remedial Program Restricted Use soil cleanup objectives (SCOs). The SCGs for groundwater are based on the NYSDEC Division of Water Technical and Operational Guidance Series (TOGS) (1.1.1) Ambient Water Quality Standards and Guidance Values and Groundwater Effluent Limitations. No SCGs currently exist for exterior soil vapor.

#### Step 6: Specify Performance or Acceptance Criteria

Specifications for this step call for: 1) giving forethought to corrective actions to improve data usability; and 2) understanding the representative nature of the sampling design. This QAPP has been designed to meet both specifications for this step. The sampling and analysis program has been developed based on a review of historical information, existing Site data collected during the Site Characterization, and knowledge of present Site conditions. The representative nature of the sampling design has been developed by discussions among professionals familiar with the Site.

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### Step 7: Develop the Plan for Obtaining Data

The overall QA objective is to develop and implement procedures for field sampling, chain-of-custody (COC), laboratory analysis, and reporting that will provide results to support the evaluation of the Site data generally consistent with National Contingency Plan (NCP) requirements. Specific procedures for sampling, COC, laboratory instrument calibration, laboratory analysis, data reporting, internal quality control, audits, preventive maintenance of field equipment, and corrective action are described in other sections of this QAPP.

The sampling plan involves a phased approach to both sampling and analysis. This provides the opportunity to evaluate and focus each data collection step to optimize the overall data collection process.

Generally, the specific RI field sampling and analysis activities to be conducted during this project which require associated QA/QC include soil, groundwater, and air sampling (i.e., soil sampling, temporary monitoring well groundwater sampling, monitoring well groundwater sampling, temporary soil vapor point sampling, ambient air quality sampling, and liquid and solid waste characterization sampling). QA/QC protocols will be implemented to ensure the PARCC parameters of the data collected during these field activities meets the objectives of the overall project. Specifically, data will be gathered or developed using procedures appropriate for the intended use of the data. The field measurements and laboratory analyses will be used to support one or more steps in the sampling described above. The PARCC parameters are further defined in Section 12.1.

The QA/QC will include laboratory method performance, field decontamination procedures, calibration and maintenance of field instruments, and QC sample collection and analysis.

A DQO summary for the sampling investigation efforts is presented in the subsequent section. The summary consists of stated DQOs relative to data uses, data types, data quantity, sampling and analytical methods, and data measurement performance criteria.

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### 5.1 Data Categories

Three data categories have been defined to address various analytical data uses and the associated QA/QC effort and methods required to achieve the desired levels of quality. These categories are:

**Screening Data**: Screening data affords a quick assessment of Site characteristics or conditions. This DQO is applicable to data collection activities that involve rapid, non-rigorous methods of analysis and quality assurance. This objective is generally applied to physical and/or chemical properties of samples, degree of contamination relative to concentration differences, and preliminary health and safety assessment.

**Screening Data with Definitive Confirmation**: Screening data allows rapid identification and quantitation, although the quantitation can be relatively imprecise. This DQO is available for data collection activities that require qualitative and/or quantitative verification of a select portion of sample findings (10% or more). This objective can also be used to verify less rigorous laboratory-based methods.

**Definitive Data**: Definitive data are generated using analytical methods such as approved USEPA reference methods. Data are analyte-specific, with confirmation of analyte identity and concentration. Methods produce raw data (e.g., chromatograms, spectra, digital values) in the form of paper printouts or computer-generated electronic files.

It is anticipated that both screening and definitive data categories will be generated during the investigation. Field parameters (e.g., turbidity, conductivity, temperature, and pH), which will be obtained during groundwater sampling for use in qualitatively interpreting other Site data, will be determined using screening techniques. Remaining parameters will be determined using definitive techniques.

For this project, only the full analytical data packages with supporting method performance data will be required from the analytical laboratory. The Level 4 data package is defined as follows:

**Level 4 – Full Reporting**: Full "CLP-type" reporting is used for those analyses that, based on intended data use, require full documentation. The Level 4 report includes analytical data as well as instrument calibration, tuning, and other raw data associated with method performance. This reporting level meets the NYSDEC ASP Superfund and Category B reporting requirements.



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The analytical methods to be used during the RI will be USEPA SW-846 methods and USEPA Compendium Method TO-15 with incorporation of the NYSDEC ASP Revision 2005 QA/QC requirements, and Category B reporting deliverables.

### 5.2 Field Investigations

As part of the RI, field investigations will be conducted to support the DQOs. Details of the field sampling investigations are described in the RI Work Plan (Section 6).

### 6. Special Training Requirements/Certification

In compliance with the Occupational Safety and Health Administration's (OSHA) final rule, "Hazardous Waste Operations and Emergency Response," 29 CFR 1910.120(e), personnel performing RI activities at the Site will have completed the requirements for OSHA 40-Hour Hazardous Waste Operations and Emergency Response (HAZWOPER) training. Persons in field supervisory positions will have also completed the additional OSHA 8-Hour Supervisory Training.

The analytical laboratory will be accredited under the New York Environmental Laboratory Approval Program (ELAP) for all methods and parameters required, and as certification is afforded under this program.

#### 7. Documentation and Records

Samples of the various media will be collected as described in the RI Work Plan. Detailed descriptions of the documentation and reporting requirements are presented below.

#### 7.1 Sample Designation System

#### 7.1.1 Sample Codes

Samples will be identified with a unique designation system that will facilitate sample tracking. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events and conditions. An alpha-numeric system is considered appropriate and will be used by field personnel to assign each sample with a unique sample identification number. The sample identification number will consist of a two-letter prefix indicating the sample type followed by numbers indicating the sample location.

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The samples types will be designated using the following codes:

- Soil Boring "SB"
- Monitoring Well "MW"
- Temporary Monitoring Well "VP"
- Soil Vapor Point "SV"
- Ambient Air Sample "AA"
- Trip Blank "TB"
- Equipment Blank "EB"

Where necessary, the code system will be supplemented to accommodate additional sample identification information. For example, the code for soil samples, temporary monitoring well groundwater samples, or soil vapor samples will include a qualifier to identify the section increment (e.g., SB-1 (2-3')).

Additional sample volumes collected for matrix spike (MS) and matrix spike duplicate (MSD) analysis will be noted on the COC forms. Trip blanks and equipment blanks will use the coding scheme noted above and a six-digit date format (e.g., an equipment blank collected on January 15, 2011 would be named EB011511). Field duplicates will be labeled as "DUP" and a six-digit date format (e.g., a field duplicate collected on January 15, 2011 would be named DUP011511). Duplicate samples will not be identified to the laboratory and the laboratory will analyze them as "blind" quality control samples. The source of the field duplicate will be noted in the field notes.

#### 7.1.2 Field Documentation

Field personnel will complete comprehensive documentation covering aspects of field sampling, field analysis, and sample COC. This documentation constitutes a record that allows reconstruction of field events to aid in the data review and interpretation process. Documents, records, and information relating to the performance of the field work will be retained in the project file.

The various forms of documentation to be maintained throughout the action include:

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- Daily Production Documentation A field notebook consisting of a waterproof, bound notebook that will contain a record of activities performed at the Site.
- Sampling Information Detailed notes will be made as to the exact sampling • location, physical observations, and weather conditions (as appropriate). Forms will be utilized for repetitive data collection, such as depth to water in wells, groundwater sampling, etc. These field forms include a Soil Sample/Core Log, Low-Flow Groundwater Sampling Form, Water-Level Measurement Form, Soil Vapor (Canister) Sample Collection Field Form, and Ambient Air (Canister) Sample Collection Field Form as applicable to a specific field task. Forms are provided in Attachment C-2. For all air samples, the initial canister vacuum and final canister vacuum must be recorded by field personnel. The initial canister vacuum should be greater than 28 inches of mercury (Hg). If the initial canister vacuum is less than 28 inches of Hg, then the canister should be returned to the laboratory and a replacement canister should be provided by the laboratory. Canister vacuums should also be recorded throughout the sampling period. Final canister vacuums of less than 1.0 to 0.1 inches of Hg upon receipt at the laboratory will result in an estimated value for detected compounds (J flag) and for non-detects (UJ flag) in accordance with NYSDEC directives. A final canister vacuum of 0.0 inches of Hg (ambient) upon receipt at the laboratory will result in an estimated value for detected compounds (J flag) and rejection for non-detects (R flag) in accordance with NYSDEC directives.
- COCs COC forms will provide the documentation of record of responsibility for sample collection, transport, and receipt by the laboratory. COC forms will be filled out at each sampling location, at a group of sampling locations, or at the end of each day of sampling by ARCADIS field personnel designated to be responsible for sample custody. In the event the samples are relinquished by the designated sampling person to other sampling or field personnel, the COC form will be signed and dated by the appropriate personnel to document the sample transfer. The original COC form will accompany the samples to the laboratory, and copies will be forwarded to the project files. A sample COC form is included in Attachment C-3.

Persons will have custody of samples when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured

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in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

To document the calibration and maintenance of field instrumentation, calibration and maintenance logs will be maintained for each piece of field equipment that is not factory-calibrated.

### 7.2 Laboratory Documentation Files

### 7.2.1 Laboratory Project Files

The laboratory will establish a file for pertinent information and communications associated with this project. The file will include correspondence, faxed information, phone logs, and COC forms. Analytical method performance data shall be retained within the laboratory in accordance with internal information and document control procedures. The laboratory will retain project files, supporting analytical method performance raw data, and data packages for a period of 10 years.

#### 7.2.2 Laboratory Logbooks

Workbooks, bench sheets, instrument logbooks, and instrument printouts will be used to trace the history of samples through the analytical process and document important aspects of the work, including the associated quality controls. As such, logbooks, bench sheets, instrument logs, and instrument printouts will be part of the permanent record of the laboratory.

Each page or entry will be dated and initialed by the analyst at the time of entry. Errors in entry will be crossed out in indelible ink with a single stroke, corrected without the use of white-out or by obliterating or writing directly over the erroneous entry, and initialed and dated by the individual making the correction. Pages of logbooks that are not used will be completed by lining out unused portions.

Information regarding the sample, analytical procedures performed, and the results of the testing will be recorded on laboratory forms or electronic information management systems and software as appropriate for the analytical method. Any analyst notes will be dated and will also identify the analyst, the instrument used, and the instrument conditions.

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Laboratory notebooks and electronic programs/calculations will be periodically reviewed by the laboratory group leaders for accuracy, completeness, and compliance to this QAPP. Entries and calculations will be verified by the laboratory group leader. If entries are correct, then the laboratory group leader will initial and date as appropriate to document the review process. Corrective action will be taken for incorrect entries before the laboratory group leader signs.

#### 7.2.3 Computer Tape and Hard Copy Storage

Electronic files and deliverables will be retained by the laboratory for not less than 10 years; hard copy data packages (or electronic copies) will also be retained for not less than 10 years.

### 7.3 Data Reporting Requirements

Data will be reported both in the field and by the analytical laboratory, as described below.

#### 7.3.1 Field Data Reporting

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the FSP and the SOPs and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

If applicable, field data forms and calculations will be processed and included in appendices to the appropriate reports (when generated). The original field logs, documents, and data reductions will be kept in the project file at ARCADIS' office.

## 7.3.2 Laboratory Data Reporting

The laboratory that analyzes the soil, groundwater, and air samples is responsible for preparing Level 4 (NYSDEC ASP Category B compliant) data packages for volatile organic compounds (VOCs), semi-volatile organic compounds (SVOCs), total cyanide, and free cyanide. Each analytical report shall include a case narrative.

Analytical reports will include, at a minimum, the following items:

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**Narrative**: Summary of activities that took place during the course of sample analysis, including the following information:

- Laboratory name and address
- Date of sample receipt
- Cross-reference of laboratory identification number to field sample identification
- Deviations from specified protocol
- Corrective actions taken

Included with the narrative will be any sample handling documents, including field and internal COC forms, air bills, and shipping tags.

**Analytical Results**: Reported according to analysis type and including the following information, as acceptable:

- Sample ID
- Laboratory ID
- Date of collection
- Date of receipt
- Date of extraction
- Date of analysis
- Analytical methods used
- Method and Reporting detection limits
- Initial and continuing calibrations
- Instrument tuning

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- Summary of quality control data: laboratory control samples, matrix spikes, laboratory duplicates, surrogate recoveries
- Method, preparation, and continuing calibration blanks
- Quantitation reports
- Chromatograms
- Extraction, preparation, digestion, and run logs
- Raw data, and
- Any other documentation required by the NYSDEC ASP Category B reports

Sample results on the report forms will be adjusted for sample volume/weight and any applicable dilutions. Soil samples will be reported on a dry weight basis. Results will not be corrected for associated blank contamination.

## 7.4 Project File

Project documentation will be placed in project files according to ARCADIS' requirements for document management. Project files typically consist of the following components:

- 1. Proposals/Agreements
- 2. Purchase Orders/Change Orders
- 3. Invoices
- 4. Project Management
- 5. Correspondence
- 6. Notes and Data
- 7. Regulatory Documents



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8. Final Reports/Presentations

### 8. Sampling Process Design

Information regarding the sampling design and rationale and associated sampling locations can be found in the RI Work Plan.

### 9. Sampling Method Requirements

Groundwater, soil, and air samples will be collected as described in the RI Work Plan, the FSP, and the SOPs. The FSP and SOPs also contain procedures that will be followed to drill and sample soil borings; drill and sample temporary monitoring wells; drill, install, and develop monitoring wells; measure water levels; collect groundwater samples; drill and sample temporary soil vapor points; collect ambient air quality samples; perform field measurements; and handle, package, and ship collected samples.

#### 10. Sample Handling and Custody Requirements

This section presents sample handling and custody requirements.

#### 10.1 Sample Containers and Preservation

Appropriate sample containers (bottles), canisters (SUMMA® canisters), preservation methods, and laboratory holding times for RI samples are provided in Table C-1.

The analytical laboratory will supply appropriate sample containers and preservatives, as necessary, or canisters. The bottles will be purchased pre-cleaned according to USEPA Office of Solid Waste and Emergency Response (OSWER) Directive 9240.05A requirements. The canisters will be cleaned and batch certified by the laboratory following the requirements of Method TO-15.

For all air samples, the initial canister vacuum and final canister vacuum must be recorded by field personnel. Canister vacuums should also be recorded throughout the sampling period. The following are special considerations for all air samples.

• The initial canister vacuum should be greater than 28 inches of Hg. If the initial canister vacuum is less than 28 inches of Hg, then the canister should be

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returned to the laboratory and a replacement canister should be provided by the laboratory.

- The final canister vacuums should be 5 inches of Hg.
- Once the canister is opened, the vacuum flow rate will be checked periodically and at least once approximately one or two hours (for ambient air samples – 8hour sampling period) after initiating sampling to make sure the vacuum is changing consistent with the allocated sampling period. If the sampling period is 30 minutes to 60 minutes (for soil vapor samples), the canister will be monitored during the entire sampling period.
- If an unexpected canister vacuum is observed, the gauge will be lightly tapped by hand to make sure it isn't stuck.
- Sampling personnel will return to the sampling location approximately two hours (ambient air samples) prior to the end of the sampling period. Soil vapor sample canister vacuums will be monitored during the entire sampling period.
  - If the canister has more than 5 inches of Hg, sampling will continue to the end of the established sampling period (8 hours for ambient air samples and 30 to 60 minutes for soil vapor samples). The canister will be closed at the end of the allocated sampling period if the vacuum is 10 inches of Hg or less. If the vacuum is greater than 10 inches of Hg at the end of the allocated sampling period, the project, task or field manager will be contacted to decide a course of action. Leaving greater than 10 inches of Hg in the canister will likely result in elevated reporting limits.
  - If the canister reaches 5 inches of Hg, the canister valve will be closed and sample collection will be terminated.
- Canisters valves will be tightened securely when sample collection is completed.

Overall, it should be noted that the analog gauges that are used on SUMMA® canisters are not extremely accurate. When in doubt, the pre-determined sampling period will be followed as a guide to when the canister should be closed. In all cases, the SUMMA® canister vacuum should NOT be allowed to go to below 5 inches of Hg.

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If the canister is received by the laboratory with zero pressure (ambient), it will be assumed that a leak occurred and the data may be considered invalid.

The field personnel will be responsible for properly labeling containers and canisters and preserving samples (as appropriate). Sample labeling procedures are discussed in Section 10.2.2.

## 10.2 Field Custody Procedures

The objective of field sample custody is to assure that samples are not tampered with from the time of sample collection through time of transport to the analytical laboratory. Persons will have "custody of samples" when the samples are in their physical possession, in their view after being in their possession, or in their physical possession and secured so they cannot be tampered with. In addition, when samples are secured in a restricted area accessible only to authorized personnel, they will be deemed to be in the custody of such authorized personnel.

Field custody documentation consists of both field logbooks and field COC forms.

#### 10.2.1 Field Logbooks

Field logbooks will provide the means of recording data collection activities performed. As such, entries will be described in as much detail as possible so that persons going to the Site could reconstruct a particular situation without reliance on memory.

Field logbooks will be bound field survey books or notebooks. Logbooks will be assigned to field personnel, but will be stored in a secure location when not in use. Each logbook will be identified by the project specific document number. The title page of each logbook will contain the following:

- Person to whom the logbook is assigned
- Logbook number
- Project name
- Project start date
- End date

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Entries into the logbook will contain a variety of information. At the beginning of each entry, the date, start time, weather, names of sampling team members present, level of personal protection being used, and the signature of the person making the entry will be entered. The names of visitors to the Site, field sampling or investigation team personnel, and the purpose of their visit will also be recorded in the field logbook.

Measurements made and samples collected will be recorded. Entries will be made in ink, and no erasures will be made. If an incorrect entry is made, the information will be crossed out with a single strike mark. Whenever a sample is collected or a measurement is made, a detailed description of the location of the station shall be recorded. The number of the photographs taken of the station, if any, will also be noted. Equipment used to make measurements will be identified, along with the date of calibration.

Samples will be collected following the sampling procedures described in the FSP and the SOPs. The equipment used to collect samples will be noted, along with the time of sampling, sample description, depth at which the sample was collected, volume, and number of containers. Sample identification numbers will be assigned prior to sample collection. Field duplicate samples, which will receive an entirely separate sample identification number, will be noted under sample description.

#### 10.2.2 Sample Labeling

Sample labels will be affixed to sample bottles at the sampling location. The following information is required on each sample label:

- Project
- Date collected
- Time collected
- Sample identification
- Sampler
- Analysis to be performed
- Preservative, as applicable



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#### 10.2.3 Field COC Forms

Completed COC forms will be required for samples. COC forms will be initiated by the sampling team in the field. The COC forms will contain the unique sample identification number, sample date and time, sample matrix, preservation (if any), and analyses required. The original COC form will accompany the samples to the laboratory. Copies of the COC will be made prior to shipment (or multiple copy forms used) for field documentation. The COC forms will remain with the samples at all times. The samples and signed COC forms will remain in the possession of the sampling crew until the samples are hand delivered to the laboratory or laboratory courier, delivered to the express carrier (e.g., FedEx), or placed in secure storage.

Sample labels will be completed for each sample using waterproof ink. The labels will be completed as described above in Section 10.2.2. The completed labels will be affixed to each sample bottle and covered with clear tape.

Whenever samples are split with a government agency or other party, a separate COC will be prepared for those samples and marked to indicate with whom the samples are being split. The person relinquishing the samples to the agency should request the representative's signature acknowledging sample receipt. If the representative is unavailable or refuses, this is noted in the "Received By" space.

#### 10.3 Management of Investigation-Derived Materials and Wastes

Management of investigation-derived materials and wastes will be performed consistent with the USEPA guidance Guide to Management of Investigation-Derived Wastes (USEPA, 1992) and following the procedures described in the ARCADIS *Investigation-Derived Waste Handling and Storage* SOP. Disposable equipment (including personal protective equipment [PPE]) and debris will be containerized and appropriately labeled during the sampling events, and will be disposed of accordingly. Purged groundwater and water generated during equipment decontamination and soil cuttings associated with drilling of soil borings will be containerized and temporarily stored on site in Department of Transportation (DOT)-approved 55-gallon steel drums, and will be disposed of properly based on analytical results. Equipment will be decontaminated, as appropriate, as discussed in the FSP and the SOP.

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#### 10.4 Packing, Handling, and Shipping Requirements

Sample packaging and shipment procedures are designed to insure that the samples will arrive at the laboratory, with the COC, intact.

Samples will be packaged for shipment as outlined below:

- Ensure that sample containers have the sample labels securely affixed to the container with clear packing tape.
- Check the caps on the sample containers to ensure that they are properly closed and sealed.
- Complete the COC form with the required sampling information and ensure that the recorded information matches the sample labels. If the designated sampler relinquishes the samples to other sampling or field personnel for packing or other purposes, the sampler will complete the COC prior to this transfer. The appropriate personnel will sign and date the COC form to document the sample custody transfer.
- Using duct tape, secure the outside drain plug (if present) at the bottom of the cooler.
- Wrap sample containers in bubble wrap or other cushioning material.
- Place bubble wrapped sample containers in Ziploc® or equivalent bags.
- Place 1 to 2 inches of cushioning material at the bottom of the cooler.
- Place the sealed sample containers into the cooler.
- Place ice in plastic bags and seal and place loosely in the cooler.
- Fill the remaining space in the cooler with cushioning material.
- Place COC forms in a plastic bag and seal. Tape the forms to the inside of the cooler lid.
- Close the lid of the cooler and secure with packing tape.

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- Wrap strapping tape around both ends of the cooler at least twice.
- Mark the cooler on the outside with the following information: shipping address, return address, "Fragile" labels, and arrows indicating "this side up." Cover the labels with clear plastic tape. Place a signed custody seal over the sample cooler lid.
- For air samples, canisters will be packaged for shipment as outlined below:
  - o Check that the canister valves are properly closed.
  - Record the initial and final canister vacuum on the COC form and on the Sample Collection Field Form.
  - Place the canisters into the shipping box.
  - o Place COC forms in a plastic bag, seal, and place in shipping box.
  - o Close the flaps of the shipping box and secure with packing tape.
  - Place a signed custody seal over the shipping box flaps.

Samples will be hand-delivered, delivered by a laboratory courier, or delivered by an express carrier within 24 hours of the time of collection. Shipments will be accompanied by the COC form identifying the contents. The original form will accompany the shipment; copies will be retained by the sampler for the sampling office records. If the samples are sent by common carrier, a bill of lading will be used. Receipts or bills of lading will be retained as part of the permanent project documentation. Commercial carriers are not required to sign off on the COC form as long as the forms are sealed inside the sample cooler and the custody seals remain intact.

Sample containers, coolers, canisters, shipping boxes, and packing materials will be provided by the analytical laboratory. The filled, labeled, and sealed containers will be placed in a cooler on ice and carefully packed to minimize the possibility of container breakage. The labeled canisters will be placed in a shipping box.



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Additional procedures for packing, handling, and shipping environmental samples are presented in the FSP and the ARCADIS *Chain-of-Custody, Handling, Packing and Shipping* SOP.

### 10.5 Laboratory Custody Procedures

Upon sample receipt, laboratory personnel will be responsible for sample custody. The original field COC form will accompany all samples requiring laboratory analysis. The laboratory will maintain internal chain of custody in accordance with laboratory policy. Samples will be kept secured in the laboratory until all stages of analysis are complete. Laboratory personnel having samples in their custody will be responsible for maintaining sample integrity.

#### 10.5.1 Sample Receipt and Storage

Immediately upon sample receipt, the laboratory sample custodian will open the cooler or shipping box, document the temperature (not applicable for air samples), and compare the contents against the field COC. If a sample container or canister is missing, a sample container is received broken, a canister appears to have leaked during shipment, the sample is in an inappropriate container, or has not been preserved by appropriate means, ARCADIS will be notified. The laboratory sample custodian will be responsible for logging the samples in, assigning a unique laboratory identification number to each sample, labeling the sample bottle or canister with the laboratory identification number, and moving the sample to an appropriate storage location to await analysis. The project name, field sample code, date sampled, date received, analysis required, storage location and date, and action for final disposition will be placed in the laboratory tracking system. Relevant custody documentation will be placed in the project file. After the sample login process has been completed, the laboratory project manager will send a sample login acknowledgement to ARCADIS to confirm the analyses to be performed.

#### 10.5.2 Sample Analysis

Samples will be organized into sample delivery groups (SDGs) by the laboratory. A SDG may contain up to 20 field samples (field duplicates, trip blanks, and equipment blanks are considered field samples for the purposes of SDG assignment). Field samples assigned to a single SDG shall be received by the laboratory over a maximum of 7 calendar days and must be processed through the laboratory (preparation,



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analysis, and reporting) as a group. A minimum of one site-specific MS/MSD pair will be included per 20 field samples.

### 10.5.3 Sample Storage Following Analysis

Samples will be maintained by the laboratory for at least one month after the final report is delivered to ARCADIS. The laboratory will be responsible for the eventual and appropriate disposal of the samples. Unused portions of the samples, sample extracts and associated wastes will be disposed of by the laboratory in accordance with applicable rules and regulations as specified in their SOP for waste disposal and federal and state requirements.

### 11. Analytical Method Requirements

This section presents analytical method requirements.

### 11.1 Field Parameters and Methods

Field analytical procedures will include the measurement of dissolved oxygen (DO), oxidation-reduction potential (ORP), pH, temperature, conductivity, turbidity, and groundwater levels. Specific field measurement protocols and instrument calibration are provided in the FSP and the SOPs.

#### 11.2 Laboratory Parameters and Methods

All soil, groundwater, and air samples will be analyzed by a New York State Department of Health (NYSDOH)-approved laboratory. The methods listed below include the analyses expected to be performed. The TestAmerica Laboratories, Inc. Quality Assurance Manual (QAM) and Standard Operating Procedures (SOPs) are provided in Attachment C-4. The Alpha Analytical, Inc. Quality Systems Manual (QSM) and SOP for Method TO-15 are provided in Attachment C-5.

Laboratory analytical requirements presented in the sub-sections below include a general summary of requirements, specifics related to each sample medium to be analyzed, and details of the methods to be used for this project. SW-846 methods, Compendium Method TO-15, and NYSDEC ASP Revision 2005 QA/QC and reporting deliverables requirements will be utilized for all analytes.

The following tables summarize general analytical requirements:

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Table	Title
Table C-1	Sample Containers, Analytical Methods, Preservation, and Holding Times
Table C-2	Quality Assurance/Quality Control Sample Summary
Tables C-3A through C-3G	Compound List and Target Reporting Limits for Water, Soil, and Air Analyses
Tables C-4A through C-4E	Project QA/QC control limits for precision and accuracy

#### 11.2.1 RI Sample Matrices

#### 11.2.1.1 Groundwater

Analyses will be performed following the methods listed in Table C-1. Analytical results for analyses will be reported in the units presented in Tables C-3A through C-3F.

### 11.2.1.2 Soil

Analyses will be performed following the methods listed in Table C-1. Analytical results will be reported as dry weight, and in the units presented in Tables C-3A through C-3F. Moisture content will be reported separately.

#### 11.2.1.3 Air

Analyses will be performed following the method listed in Table C-1. Analytical results for analyses will be reported in the units presented in Table C-3G.

#### 11.2.2 Analytical Requirements

The primary sources to describe the analytical methods to be used during the investigation are provided in USEPA SW-846 Test Methods for Evaluating Solid Waste, Third Edition as updated, USEPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, Second Edition, and NYSDEC ASP Revision 2005. Detailed information regarding QA/QC is provided in NYSDEC ASP Revision 2005.

## 12. Quality Control Requirements

This section presents quality control requirements.

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#### 12.1 Quality Assurance Indicators

The overall quality assurance objective for this QAPP is to develop and implement procedures for sampling, COC, laboratory analysis, instrument calibration, data reduction and reporting, internal quality control, audits, preventive maintenance, and corrective action, such that valid data will be generated. The PARCC parameters as related to project DQOs are discussed in this section. Specific quality control checks are discussed in Sections 12.2 and 12.3.

Quality assurance indicators are generally defined in terms of five parameters:

- a. Precision
- b. Accuracy
- c. Representativeness
- d. Completeness
- e. Comparability

Each parameter is defined below. Specific objectives for the Site actions are set forth in other sections of this QAPP as referenced below.

#### 12.1.1 Precision

Precision is the measure of reproducibility of sample results. The goal is to maintain a level of analytical precision consistent with the project objectives. To maximize precision, sampling and analytical procedures will be followed. Work for this investigation will adhere to established protocols presented in the RI Work Plan. Checks for analytical precision will include the analysis of MSDs, field duplicates, and laboratory duplicates. Checks for field measurement precision will include obtaining duplicate field measurements. Further discussion of precision quality control checks is provided in Section 12.4.

#### 12.1.2 Accuracy

Accuracy is the deviation of a measurement from the true value of a known standard. Both field and analytical accuracy will be monitored through initial and continuing

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calibration of instruments. In addition, internal standards, MSs, blank spikes, and surrogates (system monitoring compounds) will be used to assess the accuracy of the laboratory analytical data. Further discussion of these quality control samples is provided in Section 12.5.

#### 12.1.3 Representativeness

Representativeness is the degree to which sampling data accurately and precisely represent Site conditions, and is dependent on sampling and analytical variability and the variability of environmental media at the Site. The actions have been designed to assess the presence of the chemical constituents at the time of sampling. The RI Work Plan presents the rationale for sample quantities and location. This QAPP presents field sampling and laboratory analytical methodologies. The use of the prescribed field and laboratory analytical methods with associated holding times and preservation requirements are intended to provide representative data.

#### 12.1.4 Completeness

Completeness is defined as a measure of the amount of valid data obtained from an event and/or investigation compared to the total amount that was obtained. This will be determined upon final assessment of the analytical results, as discussed in Section 12.6.

#### 12.1.5 Comparability

Comparability is the degree of confidence with which one data set can be compared to another. Comparability between this investigation, and to the extent possible, with existing data will be maintained through consistent sampling and analytical methodologies set forth in the FSP, the SOPs, and this QAPP, SW-846 analytical methods, Compendium Method TO-15, with NYSDEC ASP Revision 2005 QA/QC requirements, Category B reporting deliverables, and through use of QA/QC procedures and appropriately trained personnel.



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#### 12.2 Field Quality Control Checks

#### 12.2.1 Field Measurements

To verify the quality of data using field instrumentation, duplicate measurements will be obtained and reported for field measurements. A duplicate measurement will involve obtaining measurements a second time at the same sampling location.

#### 12.2.2 Sample Containers

Certified-clean sample containers in accordance with Exhibit I of the NYSDEC ASP Revision 2005 (Eagle Picher pre-cleaned containers or equivalent) will be supplied by the laboratory. Batch certified-clean canisters in accordance with Method TO-15 will be supplied by the laboratory.

#### 12.2.3 Field Duplicates

Field duplicates will be collected from the different environmental media to verify the reproducibility of the sampling methods and potential non-homogeneity of sample locations. Field duplicate soil samples will be prepared by placing well homogenized aliquots (except samples for VOC analysis) from the same sample location into individual sample containers, which are submitted blind to the laboratory. Field duplicate soil samples for VOC analysis will constitute co-located samples rather than homogenized aliquots. In general, field duplicates will be analyzed at a 5% frequency (every 20 samples) for the chemical constituents. Table C-2 provides an estimated number of field duplicates to be collected for environmental media samples to be collected during the RI.

#### 12.2.4 Equipment Blanks

Equipment blanks are used to monitor the cleanliness of the sampling equipment and the effectiveness of the cleaning procedures. Equipment blanks will be prepared and submitted for analysis once per day per matrix when equipment decontamination is performed. Equipment blanks will be prepared by filling sample containers with analyte-free water (supplied by the laboratory) which has been routed through or over a cleaned sampling device. When dedicated sampling devices or sample containers are used to collect the samples, equipment blanks will not be necessary. Table C-2 provides an estimated number of equipment blanks for environmental media samples to be collected during the RI.



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#### 12.2.5 Trip Blanks

Trip blanks will be used to assess whether Site samples have been crosscontaminated by volatile constituents during storage and transport. Trip blanks will be analyzed at a frequency of once per day, per cooler containing samples to be analyzed for volatile organic constituents. A trip blank will be prepared by the laboratory and consist of a VOA vial filled with analyte-free water. The trip blanks will be shipped with the empty sample containers by the laboratory, will be returned with the field samples, and will remain unopened until analysis. Trip blanks will be analyzed for VOCs only. Table C-2 provides an estimated number of trip blanks for environmental media samples to be collected during the RI.

#### 12.3 Analytical Laboratory Quality Control Checks

Internal laboratory quality control checks will be used to monitor laboratory method performance and data integrity. These checks will include method blanks, MS/MSDs (not applicable for air samples), laboratory spike blanks, internal standards, surrogate compounds, calibration standards, and reference standards. Project quality control limits for precision and accuracy are identified in Tables C-4A through C-4E.

#### 12.3.1 Method Blanks

Sources of contamination in the analytical process, whether specific analyses or interferences, need to be identified, isolated, and corrected. The method blank is useful in identifying possible sources of contamination within the analytical process. For this reason, it is necessary that the method blank is initiated at the beginning of the analytical process and encompasses all aspects of the analytical work. As such, the method blank would assist in accounting for any potential contamination attributable to glassware, reagents, instrumentation, or other sources which could affect sample analysis. One method blank will be analyzed with each analytical batch associated with no more than 20 samples.

#### 12.3.2 Laboratory Control Samples

An LCS or LCS Duplicate (LCSD) consists of ASTM Type II water and, where practical, pre-cleaned sand or sodium sulfate for solid matrices, or a purchased performance testing sample. The source of the chemicals utilized for LCS spiking will be from a different supply source than the calibration standards. Where second source standards are not available, the LCS must be spiked with materials from a separate

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manufacturing lot of the standard. The LCS is generally spiked with all of the analytes of interest near the mid-point of the calibration range as defined by the method. The LCS is processed under the same sample preparation, surrogate and internal standards addition, and analytical protocols as the project samples. LCSs are analyzed at the frequency of 1 per batch of 20 samples or fewer of similar matrices. The recovery of target analytes in the LCS provides an evaluation of method performance and accuracy. Method control may be established based on the subset of compounds listed in the method. LCSDs are analyzed with some methods but are not required QA components. LCSDs are prepared and analyzed by the same protocols as the LCS. LCSD analyses provide precision evaluation of the method performance in addition to the accuracy information.

#### 12.3.3 MS/MSDs

MS/MSDs will be used to measure the accuracy of analyte recovery from the sample matrices and will be site-specific. Except for air samples, MS/MSD pairs will be analyzed at a 5% frequency (every 20 samples or once every week, whichever comes first).

When MS recoveries are outside quality control limits, associated control sample and surrogate spike recoveries will be evaluated, as applicable, to attempt to verify the reason for the deviation and determine the effect on the reported sample results. Table C-2 provides an estimated number of MS and MSD analyses for each applicable parameter.

#### 12.3.4 Laboratory Duplicates

A laboratory duplicate consists of a second aliquot selected by the laboratory from the same project sample. Selection of duplicate samples from a heterogeneous matrix requires homogenization to ensure that representative portions are analyzed. Laboratory duplicates are performed for air analyses, for which matrix spikes are not applicable. Additionally, when sample volume is limited, or for metals and general chemistry methods, a laboratory duplicate may be performed in lieu of the MSD. One sample per batch of 20 samples or fewer per matrix is analyzed as a laboratory duplicate under the above scenarios. The relative percent difference (RPD) between the results in the original and duplicate sample measure the precision of the analytical method on the actual project samples. The RPD is calculated using the same formula as the RPD for the MS/MSD and field duplicates.



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#### 12.3.5 Surrogate Spikes

Surrogates are compounds which are unlikely to occur under natural conditions that have properties similar to the analytes of interest. This type of control is primarily used for organic samples analyzed by gas chromatography/mass spectrometry (GC/MS) and GC methods and is added to the samples prior to purging or extraction. The surrogate spike is utilized to provide broader insight into the proficiency and efficiency of an analytical method on a sample-specific basis. This control reflects analytical conditions that may not be attributable to sample matrix.

If surrogate spike recoveries exceed specified quality control limits, the analytical results need to be evaluated thoroughly in conjunction with other control measures. In the absence of other control measures, the integrity of the data may not be verifiable and reanalysis of the samples with additional control may be necessary.

Surrogate spike compounds will be selected utilizing the guidance provided in the analytical methods.

#### 12.3.6 Calibration Standards

Calibration check standards analyzed within a particular analytical series provide insight regarding the instrument's stability. A calibration check standard will be analyzed at the beginning and end of an analytical series, or periodically throughout a series containing a large number of samples.

In general, calibration check standards will be analyzed after every 12 hours, or more frequently, as specified in the applicable analytical method. In analyses where internal standards are used, a calibration check standard will only be analyzed in the beginning of an analytical series. If results of the calibration check standard exceed specified tolerances, then samples analyzed since the last acceptable calibration check standard will be reanalyzed.

Laboratory instrument calibration standards will be selected utilizing the guidance provided in the analytical methods, as summarized in Section 13.

#### 12.3.7 Internal Standards

Internal standard areas and retention times will be monitored for organic analyses performed by GC/MS methods. Method-specified internal standard compounds will be



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spiked into field samples, calibration standards, and quality control samples after preparation and prior to analysis. If internal standard areas in one or more samples exceed the specified tolerances, the cause will be investigated, the instrument will be recalibrated if necessary, and affected samples will be reanalyzed.

The acceptability of internal standard performance will be determined using the guidance provided within the analytical methods.

### 12.3.8 Reference Standards/Calibration Verification

Reference standards are standards of known concentration and independent in origin from the calibration standards. The intent of reference standard analysis is to provide insight into the analytical proficiency within an analytical series. This includes preparation of calibration standards, validity of calibration, sample preparation, instrument set-up, and the premises inherent in quantitation. Reference standards will be analyzed at the frequencies specified within the analytical methods.

#### 12.4 Data Precision Assessment Procedures

Field precision is difficult to measure because of temporal variations in field parameters. However, precision will be controlled through the use of experienced field personnel, properly calibrated meters, and duplicate field measurements. Field duplicates will be used to assess precision for the entire measurement system including sampling, handling, shipping, storage, preparation, and analysis.

Laboratory data precision for organic analyses will be monitored through the use of MS/MSD and laboratory duplicates as identified in Table C-2.

The precision of data will be measured by calculation of the RPD by the following equation:

 $RPD = (A-B) \times 100$ (A+B)/2

Where:

A = Analytical result from one of two duplicate measurements

B = Analytical result from the second measurement



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Precision objectives for LCS, LCSD, MS, MSDs, and laboratory duplicate analyses are identified in the NYSDEC ASP Revision 2005 and presented in Tables C-4A through C-4E.

### 12.5 Data Accuracy Assessment Procedures

The accuracy of field measurements will be controlled by experienced field personnel, properly calibrated field meters, and adherence to established protocols. The accuracy of field meters will be assessed by review of calibration and maintenance logs.

Laboratory accuracy will be assessed via the use of MSs, surrogate spikes, internal standards, and reference standards. Accuracy will be calculated in terms of percent recovery as follows:

% Recovery =  $\underline{A-X} \times 100$ B

Where:

A = Value measured in spiked sample or standard

X = Value measured in original sample

B = True value of amount added to sample or true value of standard

This formula is derived under the assumption of constant accuracy over the original and spiked measurements. If any accuracy calculated by this formula is outside of the acceptable levels, data will be evaluated to determine whether the deviation represents unacceptable accuracy, or variable, but acceptable accuracy. Accuracy objectives for LCS, LCSD, MS, MSD, and surrogate recovery analyses are identified in the NYSDEC ASP 2005 Revision and presented in Tables C-4A through C-4E.

#### 12.6 Data Completeness Assessment Procedures

Completeness of a field or laboratory data set will be calculated by comparing the number of valid sample results generated to the total number of results generated.

Completeness = <u>number valid results</u> x 100 total number of results generated

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As a general guideline, overall project completeness is expected to be at least 90%. The assessment of completeness will require professional judgment to determine data usability for intended purposes.

## 13. Instrument/Equipment Testing, Inspection, and Maintenance Requirements

Testing and maintenance schedules have been developed for both field and laboratory instruments. A summary of the testing and maintenance activities to be performed is presented below.

### 13.1 Field Instruments and Equipment

Prior to field sampling, each piece of field equipment will be inspected to ensure that it is operational. If the equipment is not operational, it will be serviced prior to its use. Meters which require charging or batteries will be fully charged and have fresh batteries. If instrument servicing is required, it is the responsibility of the appropriate Task Manager or field personnel to follow the maintenance schedule and arrange for timely service. Field instruments will be maintained according to the manufacturers' instructions.

Logbooks will be kept for each field instrument. Each logbook will contain records of operation, maintenance, calibration, and any problems and repairs. Logbooks for each piece of equipment shall be maintained in project records. The Task Managers will review calibration and maintenance logs.

#### 13.1.1 Equipment Maintenance

Measuring and testing equipment to be used in support of the RI activities that directly affect the quality of the analytical data shall be subject to preventative maintenance measures that minimize equipment downtime. Equipment will be examined to certify that it is in operating condition. This includes checking the manufacturer's operating manual to ensure that maintenance requirements are being observed. Field notes from previous sampling events will be reviewed to ensure that any prior equipment problems are not overlooked and that any necessary repairs to equipment have been carried out.

Field equipment returned from a site will be inspected to confirm that it is in working order. The inspection will be recorded in the logbook or field notebooks, as appropriate. It will also be the obligation of the last user to record any equipment

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problems in the logbook. Non-operational field equipment will either be repaired or replaced. Appropriate spare parts will be made available for field meters.

ARCADIS-owned, subcontractor-owned, or leased equipment maintenance shall be in accordance with the manufacturer's instructions.

### 13.2 Laboratory Instruments and Equipment

Laboratory instrument and equipment documentation procedures include details of any observed problems, corrective measure(s), routine maintenance, and instrument repair (which will include information regarding the repair and the individual who performed the repair).

Preventive maintenance of laboratory equipment generally will follow the guidelines recommended by the manufacturer. A malfunctioning instrument will be repaired immediately by in-house staff or through a service call from the manufacturer.

#### 13.2.1 Instrument Maintenance

Maintenance schedules for laboratory equipment adhere to the manufacturer's recommendations. Records reflect the complete history of each instrument and specify the time frame for future maintenance. Major repairs or maintenance procedures are performed through service contracts with manufacturer or qualified contractors. Paperwork associated with service calls and preventative maintenance calls will be kept on file by the laboratory.

Laboratory Systems Managers are responsible for the routine maintenance of instruments used in the particular laboratory. Any routine preventative maintenance carried out is logged into the appropriate logbooks. The frequency of routine maintenance is dictated by the nature of samples being analyzed, the requirements of the method used, and/or the judgment of the Laboratory Systems Manager.

Major instruments are backed up by comparable (if not equivalent) instrument systems in the event of unscheduled downtime. An inventory of spare parts is also available to minimize equipment/instrument downtime.



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#### 13.2.2 Equipment Monitoring

On a daily basis, the operation of balances, incubators, ovens, refrigerators, and water purification systems will be checked and documented. Any discrepancies will be immediately reported to the appropriate laboratory personnel for resolution.

#### 14. Instrument Calibration and Frequency

This section presents instrument calibration procedures and frequency.

#### 14.1 Field Instruments and Equipment

The calibration of field instruments is governed by specific SOPs documented in the FSP for the applicable field analysis method, and such procedures take precedence over the following discussion.

Field personnel are responsible for ensuring that a master calibration/maintenance log is maintained following the procedures specified for each measuring device. Where applicable, each log will include, at a minimum, the following information:

- Name of device and/or instrument calibrated
- Device/instrument serial/identification numbers
- Calibration method
- Tolerance
- Calibration standard used
- Frequency of calibration
- Date(s) of calibration(s)
- Name of person(s) performing calibration(s)

Instruments and equipment used to gather, generate, or measure environmental data will be calibrated at the intervals specified by the manufacturer or more frequently, and in such a manner that accuracy and reproducibility of results are consistent with the

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manufacturer's specifications. In the event that an internally calibrated field instrument fails to meet calibration/checkout procedures, it will be returned to the manufacturer for service. Equipment found to be out of tolerance during the period of use shall be removed from the field and measuring and testing activities performed using the equipment shall be addressed via the corrective action system described in Section 18.3 of this QAPP.

### 14.2 Laboratory Instrument and Equipment

Instrument calibration will follow the specifications provided by the instrument manufacturer or specific analytical method used. The analytical methods for target constituents are identified separately below.

## VOCs

Equipment calibration procedures will follow SW-846 Method 8260 protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part II, Section 2.

## SVOCs

Equipment calibration procedures will follow SW-846 Method 8270 protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part II, Section 3.

## **Pesticides**

Equipment calibration procedures will follow SW-846 Method 8081 protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part II, Section 4.

## PCBs

Equipment calibration procedures will follow SW-846 Method 8082 protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part II, Section 5.

## **Metals**

Equipment calibration procedures will follow SW-846 Method 6010B and SW-846 Method 7470/7471 (mercury) protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part III, Section 1 and Section 3.

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## <u>Cyanide</u>

Equipment calibration procedures will follow SW-846 Method 9012 (total cyanide) and SW-846 Method 9016 (free cyanide) protocols and guidelines presented in NYSDEC ASP Revision 2005, Exhibit E, Part III, Section 5.

## Air Samples

Equipment calibration procedures will follow Method TO-15 protocols, the Alpha Analytical, Inc. Quality Systems Manual and SOP (Attachment C-5), and guidelines presented in NYSDEC ASP Revision 2005, Exhibit D.

## 15. Inspection/Acceptance Requirements for Supplies and Consumables

Supplies to be used in the field and laboratory will be available as needed. Preservatives and containers or canisters will be free of target chemicals and interferences. Standards will be verified against a second source standard. The laboratory will follow a "first in first out" procedure for the storage and use of consumables to minimize the risk of contamination and degradation. The various supplies and consumables required on site for field operations are listed in the FSP and field SOPs.

## 16. Data Acquisition Requirements for Non-Direct Measurements

Historical background and site usage information concerning the activities at the Site and previous investigation data collected by ARCADIS will be used as guidance in determining sampling locations for the RI.

## 17. Data Management

The purpose of data management is to ensure that the generated data are accurate and readily accessible to meet the analytical and reporting objectives of the project. The field investigations require a structured, comprehensive, and efficient program for management of data.

The data management program established for the project includes field documentation, methods for tracking and managing the data, and a system for filing Site-related information. Data management procedures will be employed to efficiently process the information collected such that the data are readily accessible and



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accurate. These procedures are described in detail in the following section which consists of five elements: 1) sample designation system; 2) field activities; 3) sample tracking and management; 4) data management system; and, 5) document control and inventory.

### 17.1 Sample Designation System

The sample designation system provides a unique sample numbering scheme that will facilitate both sample tracking and easy re-sampling of select locations to evaluate data gaps, if necessary. The sample designation system to be employed during the sampling activities will be consistent, yet flexible enough to accommodate unforeseen sampling events or conditions. A combination of letters and numbers will be used to yield a unique sample number for each field sample collected, as outlined in Section 7.1.1.

#### 17.2 Field Activities

Field activities designed to gather the information necessary to make decisions during the RI process require consistent documentation and accurate record keeping. During Site activities, standardized procedures will be used for documentation of field activities, data security, and quality assurance. These procedures are described in further detail in the following subsections.

#### 17.2.1 Field Documentation

Complete and accurate record keeping is a critical component of the field investigation activities. When interpreting analytical results and identifying data trends, investigators realize that field notes are an important part of the review and validation process. To ensure that the field investigation is thoroughly documented, several different information records, each with its own specific reporting requirements, will be maintained, including:

- Field and sampling logs
- COC forms
- Instrument calibration records, as appropriate

A description of each of these types of field documentation is provided below.

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## Field Logs

The personnel performing the field activities will keep field logs that detail observations and measurements made during the RI. Data will be recorded directly into sitededicated, bound notebooks, with each entry dated and signed. To ensure at any future date that notebook pages are not missing, each page will be sequentially numbered. Erroneous entries will be corrected by crossing out the original entry, initialing it, and then documenting the proper information. In addition, certain media sampling locations will be surveyed to accurately record their locations. The survey crew will use their own field logs and will supply the sampling location coordinates to the Database Administrator.

#### COC Forms

COC forms document sample possession from time of collection to the time of disposal. A COC form will accompany each field sample collected, and one copy of the form will be filed in the office. Field personnel will be briefed on the proper use of the COC procedure. COC procedures are included in the FSP and the ARCADIS *Chain-of-Custody, Handling, Packing, and Shipping* SOP.

#### **Instrument Calibration Records**

As part of data quality assurance procedures, field monitoring and detection equipment will be routinely calibrated. Instrument calibration ensures that equipment used is of the proper type, range, accuracy, and precision to provide data compatible with the specified requirements and desired results. Calibration procedures for the various types of field instrumentation are described in Section 14.1. In order to demonstrate that established calibration procedures have been followed, calibration records will be prepared and maintained to include, as appropriate, the following:

- Calibration date and time
- Type and identification number of equipment
- Calibration frequency and acceptable tolerances
- Identification of individual(s) performing calibration
- Reference standards used

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- Calibration data
- Information on calibration success or failure

The calibration record will serve as a written account of monitoring or detection equipment QA. Erratic behavior or failures of field equipment will be subsequently recorded in the calibration log.

#### 17.2.2 Data Security

Measures will be taken during the field investigation to ensure that samples and records are not lost, damaged, or altered. When not in use, field notebooks will be stored at the office or locked in the field vehicle. Access to these files will be limited to the field personnel who utilize them.

### 17.3 Sample Management and Tracking

A record of field documentation will be maintained to ensure the validity of data used in the Site analysis. To effectively execute such documentation, specific sample tracking and data management procedures will be used throughout the sampling program.

Sample tracking will begin with the completion of COC forms as summarized in Section 10.2.3. The completed COC forms associated with samples collected will be transmitted to the QA Officer. Copies of completed COC forms will be maintained in the office. The laboratory shall verify receipt of the samples electronically (via e-mail) on the following day.

When analytical data are received from the laboratory, the QA Officer will review the incoming analytical data packages against the information on the COCs to confirm that the correct analyses were performed for each sample and that results for samples submitted for analysis were received. Any discrepancies noted will be promptly followed-up by the QA Officer.

## 17.4 Document Control and Inventory

Project files will be maintained by ARCADIS. The types of files to be retained consist of, but are not limited to, the following:

1. Proposals/Agreements

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- 2. Purchase Orders/Change Orders
- 3. Invoices
- 4. Project Management
- 5. Correspondence
- 6. Notes and Data
- 7. Regulatory Documents
- 8. Final Reports/Presentations

#### 18. Assessment and Response Actions

Performance and systems audits may be completed in the field and laboratory during the RI as described below.

#### 18.1 Field Audits

The following field performance and systems audits will be completed during this project.

The appropriate Task Manager will monitor field performance. Field performance review summaries will contain an evaluation of field activities to verify that activities are performed according to established protocols. The QA Officer will review field reports and communicate concerns to ARCADIS' Project Manager and/or Task Managers, as appropriate. ARCADIS' QA Officer or designee will review the equipment blank and trip blank data to identify potential deficiencies in field sampling and cleaning procedures. In addition, systems audits comparing scheduled QA/QC activities from this document with actual QA/QC activities completed will be performed. The appropriate Task Manager and QA Officer will periodically confirm that work is being performed consistent with this QAPP, the RI Work Plan, the FSP, and the SOPs.

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#### 18.2 Laboratory Audits

The laboratory will perform internal audits consistent with NYSDEC ASP 2005 Revision, Exhibit E and in accordance with the New York ELAP accreditation requirements.

Internal laboratory audits are conducted by the laboratory QA manager. As part of the audit, the overall performance of the laboratory staff is evaluated and compared to the performance criteria outlined in the laboratory quality assurance manual and SOPs. The results of the audits are summarized and issued to each department supervisor, the Laboratory Manager, and the Laboratory Director. A systems audit of each laboratory is also performed by the QA manager to determine if the procedures implemented by each laboratory are in compliance with the quality assurance manual and SOPs.

In addition to the laboratory's internal audits, as participants in state and federal certification programs, the laboratory is audited by representatives of the regulatory agency issuing certification. Audits are usually conducted on an annual or biennial basis and focus on laboratory conformance to the specific program protocols for which the laboratory is seeking certification. The auditor reviews sample handling and tracking documentation, analytical methodologies, analytical supportive documentation, and final reports. The audit findings are formally documented and submitted to the laboratory for corrective action, if necessary.

ARCADIS reserves the right to conduct an on-site audit of the laboratory prior to the start of analyses for the project. Additional audits may be performed during the course of the project, as deemed necessary.

#### 18.3 Corrective Action

Corrective actions are required when field or analytical data are not within the objectives specified in this QAPP, the FSP, the SOPs, or the RI Work Plan. Corrective actions include procedures to promptly investigate, document, evaluate, and correct data collection and/or analytical procedures. Field and laboratory corrective action procedures for the actions are described below.



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#### 18.3.1 Field Procedures

When conducting the field work, if a condition is noted by the field team that would have an adverse affect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action implemented by the Field Manager or a designee, will be documented on a Corrective Action Form and reported to the appropriate ARCADIS Task Manager, QA Officer, and Project Manager.

Examples of situations that would require corrective actions are provided below:

- Protocols as defined by the QAPP, RI Work Plan, the FSP, and SOPs have not been followed.
- Equipment is not in proper working order or is not properly calibrated.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.
- Air canister valve pressure or gauges not in compliance with project requirements.

Project personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities.

18.3.2 Laboratory Procedures

In the laboratory, when a condition is noted to have an adverse affect on data quality, corrective action will be taken so as not to repeat this condition. Condition identification, cause, and corrective action taken will be documented and reported to the Project Manager and QA Officer.

Corrective action may be initiated, at a minimum, under the following conditions:

- Specific laboratory analytical protocols have not been followed.
- Protocols as defined by this QAPP have not been followed.

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- Predetermined data acceptance standards are not obtained.
- Equipment is not in proper working order or calibrated.
- Sample and test results are not completely traceable.
- QC requirements have not been met.
- Issues resulting from performance or systems audits have not been resolved.

Laboratory personnel will continuously monitor ongoing work performance in the normal course of daily responsibilities. Corrective action is initiated at a point where the problem has been identified. At whatever level this occurs (analyst, supervisor, data review, or quality control), it is brought to the attention of the laboratory QA Officer and, ultimately, the Laboratory Director. Final approval of any action deemed necessary is subject to the approval of the Laboratory Director.

Any corrective action deemed necessary based on system or performance audits or the results of data review will be implemented. The corrective action may include sample re-extraction, re-preparation, re-analysis, cleanup, dilutions, matrix modifications, or other activities.

## 19. Reporting

This section presents reporting requirements.

#### 19.1 Internal Reporting

The analytical laboratory will submit analytical reports to ARCADIS for review. The reports will then be submitted to the data validator for review. Supporting data (i.e., historic data, related field or laboratory data) will also be reviewed to evaluate data quality, as appropriate. ARCADIS' QA Officer will incorporate results of the data validation reports and assessments of data usability into a summary report (if required) that will be submitted to ARCADIS' Project Manager and appropriate Task Managers. If required, this report will be filed in the project file at ARCADIS' office and will include the following:

1. Assessment of data accuracy, precision, and completeness for both field and laboratory data.

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- 2. Results of the performance and systems audits.
- 3. Significant QA/QC problems, solutions, corrections, and potential consequences.
- 4. Analytical data validation report.

#### 19.2 RI Reporting

Upon sample transport to the laboratory, a copy of the COC and laboratory sample login documentation will be forwarded to ARCADIS' Project Manager. Upon receipt of the analytical data package from the laboratory, ARCADIS' QA Officer or designee will determine if the data has met the required data quality objectives. The analytical data package will be submitted to ARCADIS' Project Manager and the analytical data will be incorporated into the RI Report in a tabulated format.

### 20. Data Reduction and Review

After field and laboratory data are obtained, the data will be subject to the following:

- 1. Reduction, or manipulation mathematically, or otherwise into meaningful and useful forms.
- 2. Review.
- 3. Data validation.
- 4. Organization, interpretation, and reporting.

#### 20.1 Field Data Reduction and Review

#### 20.1.1 Field Data Reduction

Information collected in the field through visual observation, manual measurement, and/or field instrumentation will be recorded in field notebooks or data sheets, and/or on forms. Such data will be reviewed by the appropriate Task Manager for adherence to the RI Work Plan, the FSP, the SOPs, and this QAPP and for consistency. Concerns identified as a result of this review will be discussed with the field personnel, corrected if possible, and, as necessary, incorporated into the data evaluation process.

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#### 20.1.2 Field Data Review

Field data calculations, transfers, and interpretations will be conducted by the field personnel and reviewed for accuracy by the appropriate Task Manager and the QA Officer. Logs and documents will be checked for:

- 1. General completeness.
- 2. Readability.
- 3. Usage of appropriate procedures.
- 4. Appropriate instrument calibration and maintenance.
- 5. Reasonableness in comparison to present and past data collected.
- 6. Correct sample locations.
- 7. Correct calculations and interpretations.

#### 20.2 Laboratory Data Reduction and Review

#### 20.2.1 Laboratory Data Reduction

The calculations used for data reduction will be specified in each of the analytical methods referenced previously. Whenever possible, analytical data will be transferred directly from the instrument to a computerized data system. Raw data not amenable to electronic management will be entered into permanently bound laboratory notebooks. The data entered are sufficient to document factors used to arrive at the reported value.

Concentration calculations for chromatographic analyses will be based on response factors. Quantitation will be performed using either internal or external standards.

Inorganic analyses will be based on regression analysis. Regression analysis is used to fit a curve through the calibration standard data. The sample concentrations will be calculated using the resulting regression equations.

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Non-aqueous values will be reported on a dry-weight basis. Values will not be corrected for blank contamination.

### 20.2.2 Laboratory Data Review

Data will be subject to multi-level review by the laboratory. The group leader will review data reports prior to release for final data report generation. The QA manager will review approximately 10% of the final data reports and the Laboratory Project Manager will review a cross-section of the final data reports prior to shipment to ARCADIS.

If discrepancies or deficiencies exist in the analytical results, then corrective action will be taken, as discussed in Section 18.3. Deficiencies discovered as a result of internal data review, as well as the corrective actions to be used to rectify the situation, will be documented on a Corrective Action Form and summarized in the case narrative.

#### 20.2.3 Data Validation and Verification

Data generated for site investigation purposes will be subjected to the data validation and verification procedures outlined in Section 21. Data generated for waste characterization and disposal purposes will not be reviewed unless anomalous results are observed.

## 21. Data Validation and Verification

Data validation entails a review of the quality control data and the raw data to verify that the laboratory was operating within required limits, the analytical results were correctly transcribed from the instrument read outs, and which, if any, environmental samples were related to any out-of-control quality control samples. The objective of data validation is to identify any questionable or invalid laboratory measurements.

ARCADIS will validate data generated and produce a NYSDEC DUSR using this QAPP, analytical method performance criteria, laboratory control limits, NYSDEC ASP Revision 2005 requirements, the USEPA's National Functional Guidelines (USEPA, 2004; USEPA, 2008), and USEPA Region 2 SOPs for data validation. These procedures and criteria may be modified as necessary to address project-specific and method-specific criteria, control limits, and procedures. Data validation will consist of data screening, checking, reviewing, editing, and interpretation to document analytical data quality and to determine whether the quality is sufficient to meet the DQOs.

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The data validator will verify that reduction of laboratory measurements and laboratory reporting of analytical parameters is in accordance with the procedures specified for each analytical method and/or as specified in this QAPP and NYSDEC ASP Revision 2005. Deviations from the analytical method or any special reporting requirements apart from that specified in this QAPP will be detailed in the analytical reports.

Upon receipt of laboratory data, the following procedures will be executed by the data validator:

- Evaluate completeness of data package.
- Verify that field COC forms were completed and that samples were handled properly.
- Verify that holding times were met for each parameter. Holding time exceedences, should they occur, will be documented. Data for samples exceeding holding time requirements will be flagged as either estimated or rejected. The decision as to which qualifier is more appropriate will be made on a case-by-case basis. In general, if the holding time is exceeded by less than two times the EPA recommended holding time, the data will be qualified as estimated.
- Verify that parameters were analyzed according to the methods specified.
- Verify compliance with canister pressure requirements and tracking.
- Review QA/QC data (i.e., make sure duplicates, blanks, and spikes were analyzed on the required number of samples, as specified in the method; verify that accuracy and precision of quality control data are acceptable).
- For VOC and SVOC analyses, review blank contamination, mass spectrometer tuning, calibration information [initial and continuing calibrations], surrogates/system monitoring compounds, internal standard performance, MS/MSD analysis, LCS/LCSD analysis, field duplicate analysis, compound identification, overall system performance, and raw data.
- For pesticide and PCB analyses, review blank contamination, calibration information [initial and continuing calibrations], surrogates/system monitoring

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compounds, MS/MSD analysis, LCS/LCSD analysis, field duplicate analysis, compound identification, overall system performance, and raw data.

- For metals and cyanide analyses, review blank contamination, calibration information [initial and continuing calibrations], MS/laboratory duplicate analysis, field duplicate analysis, LCS analysis, overall system performance, and raw data.
- Prepare a Data Usability Summary Report (DUSR).
- Investigate anomalies identified during review. When anomalies are identified, they will be discussed with the Project Manager and/or Laboratory Manager, as appropriate.
- If data appears suspect, further data evaluation will be performed to investigate the specific data of concern. This review may include evaluation of verification of calculations and other reviews of raw data.

Deficiencies discovered as a result of the data review will be documented and submitted in the form of a written validation report addressing the following topics as applicable to each method:

- Assessment of the data package
- Description of any protocol deviations
- Summary of the QC failures observed
- Assessment of any compromised data
- Summary of the qualified data

It should be noted that qualified results do not necessarily invalidate data. The goal to produce the best possible data does not necessarily mean producing data without quality control qualifiers. Data qualified as estimated will be utilized for site evaluation. Rejected data will not be used.



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Resolution of any issues regarding laboratory performance or deliverables will be handled between the laboratory and the data validator. Suggestions for re-sampling or reanalysis may be made by ARCADIS' QA Officer at this point.

DUSRs will be kept in the project file at ARCADIS' office.

#### 22. Reconciliation with User Requirements

The data results will be examined to determine the performance that was achieved for each data usability criteria. The performance will then be compared with the project objectives and DQOs. Deviations from objectives will be noted. Additional action may be warranted when performance does not meet performance objectives for critical data. Options for corrective action relating to incomplete information, questionable results or inconsistent data, may include any or all of the following:

- Retrieval of missing information
- Request for additional explanation or clarification
- Re-extraction or reanalysis of sample (when appropriate)
- Recalculation or reinterpretation of results by the laboratory

These actions may improve the data quality, reduce uncertainty, and may eliminate the need to qualify or reject data.

If these actions do not improve the data quality to an acceptable level, the following additional actions may be taken:

- Extrapolation of missing data from existing data points
- Use of historical Site use information
- Evaluation of the critical/non-critical nature of the sample

If the data gap cannot be resolved by these actions, an evaluation of the data bias and potential for false negatives and positives can be performed. If the resultant uncertainty level is unacceptable, the following action must be taken:

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• Additional sample collection and analysis

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#### 23. References

- New York State Department of Environmental Conservation (NYSDEC). 2010. DER-10 Technical Guidance for Site Investigation and Remediation. May 2010.
- U.S. Environmental Protection Agency (USEPA). 2008. USEPA Contract Laboratory Program National Functional Guidelines for Superfund Organic Methods Data Review, USEPA-540-R-08-01. June 2008.
- U.S. Environmental Protection Agency (USEPA). 2006. Guidance on Systematic Planning Using the Data Quality Objectives Process, EPA QA/G-4, EPA/240/B-06/001. February 2006.
- U.S. Environmental Protection Agency (USEPA). 2004. USEPA Contract Laboratory Program National Functional Guidelines for Inorganic Data Review, EPA 540-R-04-004. October 2004.
- U.S. Environmental Protection Agency (USEPA). 2002. Guidance for Quality Assurance Project Plans, EPA QA/G-5, EPA/240/R-02/009. December 2002.
- U.S. Environmental Protection Agency (USEPA). 1992. Guide to Management of Investigation-Derived Wastes, Publication 9345.3-03FS. January 1992.

Table C-1. Summary of Sample Containers, Analytical Methods, Preservation, and Holding Times, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

Parameter	Method <sup>1</sup>	Sample Container	Preservation	Holding Time
Soil				
VOCs	8260B	1 x 2-oz glass jar with Teflon®-lined septa	Cool 4° C	12 Days VTSR
SVOCs	8270C	1 x 8-oz glass jar with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TCL Pesticides	8081A	1 x 8-oz glass jar with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TCL PCBs	8082	1 x 8-oz glass jar with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TAL Metals	6010B/7471A	1 x 8-oz glass jar	Cool 4° C	178 Days VTSR; Mercury 26 Days VTSR
Free Cyanide	9016	1 x 8-oz glass jar	Cool 4° C	12 Days VTSR
Total Cyanide	9012B	1 x 8-oz glass jar	Cool 4° C	12 Days VTSR
Water				
VOCs	8260B	3 x 40-mL glass vial with Teflon®-lined septa	Cool 4° C, HCl to pH <2	12 Days VTSR
SVOCs	8270C	2 x 1-L amber glass bottle with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TCL Pesticides	8081A	2 x 1-L amber glass bottle with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TCL PCBs	8082	2 x 1-L amber glass bottle with Teflon®-lined cap	Cool 4° C	5 Days VTSR to Extraction, 40 Days to Analysis
TAL Metals	6010B/7470	1 x 500-mL plastic bottle	Cool 4° C, HNO <sub>3</sub> to pH <2	178 Days VTSR; Mercury 26 Days VTSR
Total Cyanide	9012B	1 x 500-mL plastic bottle	Cool 4° C, NaOH to pH >12	12 Days VTSR
Air				
TO-15 VOCs Expanded	TO-15 <sup>2</sup>	One (1) 6-L SUMMA® Canister		30 Days
1	USEPA. Office of Soli	d Waste. Test Methods for Evaluating Solid Waste (SW-8	346).	
2	USEPA. Compendium	n of Methods for the Determination of Toxic Organic Comp	oounds in Ambient Air - Second E	dition.
VOCs	Volatile Organic Com	pounds.		
SVOCs	Semi-Volatile Organic	Compounds.		
PCBs	Polychlorinated Biphe	nyls.		
TCL	Target Compound Lis	t.		
TAL	Target Analyte List.			
oz	Ounce.			
mL	Milliliter.			
L	Liter.			
С	Celsius.			
HCI	Hydrochloric Acid.			
HNO <sub>3</sub>	Nitric Acid.			
NaOH	Sodium Hydroxide.			
VTSR	Validated Time of Sar			

Table C-2. Quality Assurance/Quality Control Sample Summary, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

Parameter	Estimated Environmental	Field QC Samples				Laboratory QC Samples				Total		
	Sample Quantity	Equipmer	nt Blanks	Trip B	lanks	Field Du	plicates	Matrix	Spike	Matrix Spike Duplica	ate or Lab Duplicate	
		Frequency	Quantity	Frequency	Quantity	Frequency	Quantity	Frequency	Quantity	Frequency	Quantity	
Soil <sup>1</sup>												
Volatile Organic Compounds (SW-846 8260B)	56	1/Day	20	1/Cooler	20	1/20	3	1/20	3	1/20	3	105
Semi-Volatile Organic Compounds (SW-846 8270C)	56	1/Day	20	NA	NA	1/20	3	1/20	3	1/20	3	85
TCL Pesticides (SW-846 8081A)	11	1/Day	11	NA	NA	1/20	1	1/20	1	1/20	1	25
TCL Polychlorinated Biphenyls (SW-846 8082)	56	1/Day	20	NA	NA	1/20	3	1/20	3	1/20	3	85
TAL Metals (SW-846 6010B/7471A)	56	1/Day	20	NA	NA	1/20	3	1/20	3	1/20	3	85
Free Cyanide (SW-846 9016)	56	1/Day	20	NA	NA	1/20	3	1/20	3	1/20	3	85
Total Cyanide (SW-846 9012B)	56	1/Day	20	NA	NA	1/20	3	1/20	3	1/20	3	85
Water												
Volatile Organic Compounds (SW-846 8260B)	34	1/Day	10	1/Cooler	10	1/20	2	1/20	2	1/20	2	60
Semi-Volatile Organic Compounds (SW-846 8270C)	34	1/Day	10	NA	NA	1/20	2	1/20	2	1/20	2	50
TCL Pesticides (SW-846 8081A)	3	1/Day	1	NA	NA	1/20	1	1/20	1	1/20	1	7
TCL Polychlorinated Biphenyls (SW-846 8082)	3	1/Day	1	NA	NA	1/20	1	1/20	1	1/20	1	7
TAL Metals (SW-846 6010B/7470)	16	1/Day	4	NA	NA	1/20	1	1/20	1	1/20	1	23
Total Cyanide (SW-846 9012B)	16	1/Day	4	NA	NA	1/20	1	1/20	1	1/20	1	23
Air												
TO-15 VOCs Expanded	5	NA	NA	NA	NA	1/20	1	NA	NA	NA	NA	6

1 Sample Quantity is an approximation; the final sample quantity will be determined in the field based on field conditions and observations.

TCL Target Compound List.

 TAL
 Target Analyte List.

 QC
 Quality Control

 NA
 Not Applicable

Table C-3A. Compound List and RLs for Water and Soil VOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	eporting Limits	
Compound	Water (ug/L)	Soil (ug/kg)	
Dichlorodifluoromethane	5	5	
Chloromethane	5	5	
Vinyl Chloride	5	5	
Bromomethane	5	5	
Chloroethane	5	5	
Trichlorofluoromethane	5	5	
1,1-Dichloroethene	5	5	
1,1,2-Trichloro-1,2,2-trifluoroethane	5	5	
Acetone	10	25	
Carbon Disulfide	5	5	
Methyl Acetate	5	5	
Methylene Chloride	5	20	
trans-1,2-Dichloroethene	5	5	
Methyl tert-Butyl Ether	5	5	
1,1-Dichloroethane	5	5	
cis-1,2-Dichloroethene	5	5	
2-Butanone	10	25	
Chloroform	5	5	
1,1,1-Trichloroethane	5	5	
Cyclohexane	5	5	
Carbon Tetrachloride	5	5	
Benzene	5	5	
1,2-Dichloroethane	5	5	
Trichloroethene	5	5	
Methylcyclohexane	5	5	
1,2-Dichloropropane	5	5	
Bromodichloromethane	5	5	
cis-1,3-Dichloropropene	5	5	
4-Methyl-2-pentanone	10	25	
Toluene	5	5	
trans-1,3-Dichloropropene	5	5	
1,1,2-Trichloroethane	5	5	
Tetrachloroethene	5	5	
2-Hexanone	10	25	
Dibromochloromethane	5	5	
1,2-Dibromoethane	5	5	
Chlorobenzene	5	5	
Ethylbenzene	5	5	
Xylenes (total)	5	10	
Styrene	5	5	
Bromoform	5	5	
Isopropylbenzene	5	5	
1,1,2,2-Tetrachloroethane	5	5	
1,1,∠,∠-1 511 a011101 0511 a115	5	5	

See last page for footnotes.

Table C-3A. Compound List and RLs for Water and Soil VOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	_	
Compound	Water (ug/L)	Soil (ug/kg)	
1,3-Dichlorobenzene	5	5	
1,4-Dichlorobenzene	5	5	
1,2-Dichlorobenzene	5	5	
1,2-Dibromo-3-chloropropane	5	10	
1,2,4-Trichlorobenzene	5	5	
1,4-Dioxane	50	200	
n-Butylbenzene	5	5	
n-Propylbenzene	5	5	
sec-Butylbenzene	5	5	
tert-Butylbenzene	5	5	
1,2,4-Trimethylbenzene	5	5	
1,3,5-Trimethylbenzene	5	5	

1. Compound list is a combination of USEPA Contract Laboratory Program Statement of Work OLM04.2 TCL for Volatile Compounds and compounds listed in 6 NYCRR Subpart 375-6.

 RLs
 Reporting Limits.

 VOC
 Volatile Organic Compound.

 TCL
 Target Compound List.

 ug/L
 Micrograms per liter.

Table C-3B. Compound List and RLs for Water and Soil SVOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	eporting Limits	
	Water	Soil	
Compound	(ug/L)	(ug/kg)	
Benzaldehyde	10	330	
Phenol	10	330	
pis(2-chloroethyl)ether	1	33	
2-Chlorophenol	10	330	
2-Methylphenol	10	330	
2,2'-oxybis(1-Chloropropane)	10	330	
Acetophenone	10	330	
4-Methylphenol	10	330	
N-Nitroso-di-n-propylamine	1	33	
Hexachloroethane	1	33	
Nitrobenzene	1	33	
sophorone	10	330	
2-Nitrophenol	10	330	
2,4-Dimethylphenol	10	330	
bis(2-chloroethoxy)methane	10	330	
2,4-Dichlorophenol	10	330	
Naphthalene	10	330	
4-Chloroaniline	10	330	
Hexachlorobutadiene	2	67	
Caprolactum	10	330	
4-Chloro-3-methylphenol	10	330	
	10	330	
2-Methylnapthalene	10	330	
Hexachlorocyclopentadiene			
2,4,6-Trichlorophenol	10	330	
2,4,5-Trichlorophenol	10	330	
1,1'-Biphenyl	10	330	
2-Chloronaphthalene	10	330	
2-Nitroaniline	20	670	
Dimethylphthalate	10	330	
2,6-Dinitrotoluene	2	67	
Acenaphthylene	10	330	
3-Nitroaniline	20	670	
Acenaphthene	10	330	
2,4-Dinitrophenol	30	1000	
1-Nitrophenol	30	1000	
Dibenzofuran	10	330	
2,4-Dinitrotoluene	2	67	
Diethylphthalate	10	330	
Fluorene	10	330	
4-Chlorophenyl-phenyl ether	10	330	
4-Nitroaniline	20	670	
4,6-Dinitro-2-methylphenol	30	1000	
N-Nitrosodiphenylamine	10	330	

See last page for footnotes.

Table C-3B. Compound List and RLs for Water and Soil SVOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	eporting Limits	
Compound	Water (ug/L)	Soil (ug/kg)	
4-Bromophenyl-phenylether	10	330	
Hexachlorobenzene	1	33	
Atrazine	10	330	
Pentachlorophenol	30	1000	
Phenanthrene	10	330	
Anthracene	10	330	
Carbazole	10	330	
Di-n-butylphthalate	10	330	
Fluoranthene	10	330	
Pyrene	10	330	
Butylbenzylphthalate	10	330	
3,3'-Dichlorobenzidine	20	670	
Benzo(a)anthracene	1	33	
Chrysene	10	330	
bis(2-ethylhexyl)phthalate	10	330	
Di-n-octylphthalate	10	330	
Benzo(b)fluoranthene	1	33	
Benzo(k)fluoranthene	1	33	
Benzo(a)pyrene	1	33	
Indeno(1,2,3-cd)pyrene	1	33	
Dibenzo(a,h)anthracene	1	33	
Benzo(g,h,i)perylene	10	330	
3-Methylphenol	10	330	
Hexachlorobenzene	10	330	

1. Compound list is a combination of USEPA Contract Laboratory Program Statement of Work OLM04.2 TCL for Semi-Volatile Compounds and compounds listed in 6 NYCRR Subpart 375-6.

RLs Reporting Limits.

SVOC Semi-Volatile Organic Compound.

TCL Target Compound List.

ug/L Micrograms per liter.

Table C-3C. Compound List and RLs for Water and Soil Pesticide Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	eporting Limits	
Compound	Water (ug/L)	Soil (ug/kg)	
4,4'-DDD	0.05	6.7	
4,4'-DDE	0.05	6.7	
4,4'-DDT	0.05	6.7	
Aldrin	0.05	6.7	
alpha-BHC	0.05	6.7	
alpha-Chlordane	0.05	6.7	
beta-BHC	0.05	6.7	
delta-BHC	0.05	6.7	
Dieldrin	0.05	6.7	
Endosulfan I	0.05	6.7	
Endosulfan II	0.05	6.7	
Endosulfan sulfate	0.05	6.7	
Endrin	0.05	6.7	
Endrin aldehyde	0.05	6.7	
Endrin ketone	0.05	6.7	
gamma-BHC (Lindane)	0.05	6.7	
gamma-Chlordane	0.05	6.7	
Heptachlor	0.05	6.7	
Heptachlor epoxide	0.05	6.7	
Methoxychlor	0.05	6.7	
Toxaphene	0.5	67	

1. Compound list refers to USEPA Contract Laboratory Program Statement of Work OLM04.2 TCL for Pesticides.

RLs Reporting Limits.

TCL Target Compound List.

ug/L Micrograms per liter.

Table C-3D. Compound List and RLs for Water and Soil PCB Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

Compound	Laboratory Reporting Limits				
	Water (ug/L)	Soil (ug/kg)			
Aroclor-1016	0.5	67			
Aroclor-1221	0.5	67			
Aroclor-1232	0.5	67			
Aroclor-1242	0.5	67			
Aroclor-1248	0.5	67			
Aroclor-1254	0.5	67			
Aroclor-1260	0.5	67			

1. Compound list refers to USEPA Contract Laboratory Program Statement of Work OLM04.2 TCL for Aroclors.

RLs Reporting Limits.

TCL Target Compound List.

ug/L Micrograms per liter.

Table C-3E. Analyte List and RLs for Water and Soil Metals Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory R	eporting Limits	_
Analyte	Water (ug/L)	Soil (mg/kg)	
Aluminum	200	10	
Antimony	10	0.5	
Arsenic	5	0.25	
Barium	200	10	
Beryllium	2	0.1	
Cadmium	5	0.25	
Calcium	5000	250	
Chromium	10	0.5	
Cobalt	50	2.5	
Copper	25	1.25	
Iron	150	7.5	
Lead	5	0.25	
Magnesium	5000	250	
Manganese	15	0.75	
Mercury	0.2	0.033	
Nickel	40	2	
Potassium	5000	250	
Selenium	10	0.5	
Silver	10	0.5	
Sodium	5000	250	
Thallium	10	0.5	
Vanadium	50	2.5	
Zinc	30	1.5	

1. Analyte list refers to USEPA Contract Laboratory Program Statement of Work ILM04.1 TAL for Metals.

RLs Reporting Limits.

TAL Target Analyte List.

ug/L Micrograms per liter.

mg/kg Milligrams per kilogram.

Table C-3F. RLs for Water and Soil Cyanide Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory R	eporting Limits
Analyte	Water (ug/L)	Soil (mg/kg)
Free Cyanide		2.3
Total Cyanide	10	0.5

RLs Reporting Limits. ug/L Micrograms per liter.

mg/kg Milligrams per kilogram.

Table C-3G. Compound List and RLs for Soil Vapor and Ambient Air VOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Ro	eporting Limits	
Compound	(ppbv)	(ug/m <sup>3</sup> )	
Acetone	0.5	1.18	
Benzene	0.2	0.64	
Bromodichloromethane	0.2	1.34	
Vinyl bromide (Bromoethene)	0.2	0.87	
Bromoform	0.2	2.07	
Bromomethane (Methyl bromide)	0.2	0.78	
1,3-Butadiene	0.2	0.44	
2-Butanone (Methyl ethyl ketone)	0.2	0.59	
Carbon disulfide	0.2	0.62	
Carbon tetrachloride	0.2	1.26	
Chlorobenzene	0.2	0.92	
Chloroethane	0.2	0.53	
Chloroform	0.2	0.98	
Chloromethane (Methyl chloride)	0.2	0.41	
3-Chloropropene (allyl chloride)	0.2	0.63	
2-Chlorotoluene (o-Chlorotoluene)	0.2	1.03	
Cyclohexane	0.2	0.69	
Dibromochloromethane	0.2	1.7	
1,2-Dibromoethane	0.2	1.54	
1,2-Dichlorobenzene	0.2	1.2	
	0.2	1.2	
1,3-Dichlorobenzene			
1,4-Dichlorobenzene	0.2	1.2	
Dichlorodifluoromethane (Freon 12)	0.2	0.99	
1,1-Dichloroethane	0.2	0.81	
1,2-Dichloroethane	0.2	0.81	
1,1-Dichloroethene	0.2	0.79	
cis-1,2-Dichloroethene	0.2	0.79	
trans-1,2-Dichloroethene	0.2	0.79	
1,2-Dichloropropane	0.2	0.92	
cis-1,3-Dichloropropene	0.2	0.91	
trans-1,3-Dichloropropene	0.2	0.91	
1,2-Dichlorotetrafluoroethane (Freon 114)	0.2	1.4	
1,4-Dioxane	0.2	0.72	
Ethylbenzene	0.2	0.87	
4-Ethyltoluene (p-Ethyltoluene)	0.2	0.98	
n-Heptane	0.2	0.82	
Hexachlorobutadiene	0.2	2.13	
n-Hexane	0.2	0.7	
2-Hexanone (Methyl Butyl Ketone)	0.2	0.82	
Isopropyl Alcohol	0.5	1.23	
Methylene chloride	0.5	1.73	
4-Methyl-2-pentanone (MIBK)	0.2	0.82	
Methyl tert-butyl ether (MTBE)	0.2	0.72	
Styrene	0.2	0.85	
Tertiary butyl alcohol (TBA)	0.2	0.61	
1,1,2,2-Tetrachloroethane	0.2	1.37	
Tetrachloroethene (PCE)	0.2	1.36	
Toluene	0.2	0.75	
1,2,4-Trichlorobenzene	0.2	1.48	
1,1,1-Trichloroethane	0.2	1.09	
1,1,2-Trichloroethane	0.2	1.09	
1,1,2-Trichloro-1,2,2-trifluoroethane (Freon 113)	0.2	1.53	
Trichloroethene (TCE)	0.2	1.07	
Trichlorofluoromethane (Freon 11)	0.2	1.12	
1,2,4-Trimethylbenzene	0.2	0.98	

See footnotes on last page.

Table C-3G. Compound List and RLs for Soil Vapor and Ambient Air VOC Analysis, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	Laboratory Re	eporting Limits	
Compound	(ppbv)	(ug/m³)	
1,3,5-Trimethylbenzene	0.2	0.98	
2,2,4-Trimethylpentane	0.2	0.93	
Vinyl Chloride	0.2	0.51	
m&p-Xylenes	0.4	1.72	
o-Xylene	0.2	0.87	
Additional Compounds			
1,2,3-Trimethylbenzene	0.2	0.98	
Naphthalene	0.2	1.05	
1-Methylnaphthalene	2.5	14.5	
2-Methylnaphthalene	2.5	14.5	
1,2,4,5-Tetramethylbenzene	2.5	13.7	
Indene	0.2	0.95	
Indane	0.2	0.97	
Thiopene	0.2	0.69	

RLs Reporting Limits.

ppbv Parts per billion by volume.

ug/m<sup>3</sup> Micrograms per cubic meter.

Table C-4A. VOC Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	<u> </u>	% Recovery		RPD	
	Water	Soil/Sediment	Water	Soil/Sediment	
/OC Surrogate Recovery Limits					
Compound <sup>1</sup>					
1,2-Dichloroethane-d4	66-137	64-126	NA	NA	
Toluene-d8	71-126	71-125	NA	NA	
1-Bromofluorobenzene	73-120	72-126	NA	NA	
LCS (Blank) and Matrix Spike Recovery and RPD Li	mits				
Compound					
1,1,1-Trichloroethane	73-126	77-121	15	20	
1,1,2,2-Tetrachloroethane	70-126	80-120	15	20	
1,1,2-Trichloro-1,2,2-trifluoroethane	60-140	60-140	20	20	
1,1,2-Trichloroethane	76-122	78-122	15	20	
1,1-Dichloroethane	71-129	79-126	20	20	
I,1-Dichloroethene	65-138	65-153	16	20	
I,2,4-Trichlorobenzene	70-122	64-120	20	20	
,2,4-Trimethylbenzene	76-121	74-120	20	20	
,2-Dibromo-3-Chloropropane	56-134	63-124	15	20	
,2-Dichlorobenzene	77-120	75-120	20	20	
I,2-Dichloroethane	75-127	77-122	20	20	
1,2-Dichloropropane	76-120	75-124	20	20	
I,3,5-Trimethylbenzene	77-121	74-120	20	20	
I,3-Dichlorobenzene	77-120	74-120	20	20	
I,4-Dichlorobenzene	75-120	73-120	20	20	
I,4-Dioxane					
2-Butanone (MEK)	65-127	70-134	15	20	
2-Hexanone	56-142	59-130	15	20	
4-Methyl-2-pentanone (MIBK)	71-124	65-133	13	20	
Acetone	66-128	61-137	15	20	
Benzene	36-150	79-127	15	20	
Bromodichloromethane	59-134	80-122	15	20	
Bromoform	72-134	68-126	15	20	
Bromomethane	72-120	37-149	25	20	
Carbon disulfide	75-125	64-131	15	20	
Carbon tetrachloride	69-136	75-135	15	20	
Chlorobenzene	73-127	76-124	20	20	
Chloroethane	49-142	69-135	15	20	

See footnotes on last page.

Table C-4A. VOC Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	% R	ecovery		RPD
	Water	Soil/Sediment	Water	Soil/Sediment
LCS (Blank) and Matrix Spike Recovery and RPD Limits				
Compound				
Chloroform	74-124	80-118	15	20
Chloromethane	74-124	63-127	15	20
cis-1,2-Dichloroethene	70-130	81-117	20	20
cis-1,3-Dichloropropene	80-122	82-120	15	20
Cyclohexane	33-157	70-130	20	20
Dibromochloromethane	77-123	76-125	15	20
Dichlorodifluoromethane	77-122	57-142	20	20
Ethylbenzene	60-140	80-120	20	20
Isopropylbenzene	57-140	72-120	20	20
Methyl acetate	71-125	60-140	35	20
Methyl tert-butyl ether	64-127	63-125	37	20
Methylcyclohexane	60-140	60-140	20	20
Methylene Chloride	57-132	61-127	15	20
n-Butylbenzene	71-128	70-120	15	20
N-Propylbenzene	77-120	70-130	15	20
sec-Butylbenzene	74-127	74-120	15	20
Styrene	70-130	80-120	20	20
tert-Butylbenzene	75-123	73-120	15	20
Tetrachloroethene	74-122	74-122	20	20
Toluene	70-122	74-128	15	20
trans-1,2-Dichloroethene	73-127	78-126	20	20
trans-1,3-Dichloropropene	72-123	73-123	15	20
Trichloroethene	74-123	77-129	16	20
Trichlorofluoromethane	62-152	64-146	20	20
Vinyl chloride	65-133	61-133	15	20
Xylenes, Total	76-122	70-130	16	20

1 The recovery limits for any of the compounds listed above may be expanded at any time during the period of performance if USEPA determines that the limits are too restrictive.

See NYSDEC Revision 2005 for additional method performance criteria.

VOC Volatile Organic Compound.

- QC Quality Control.
- LCS Laboratory Control Sample or Blank Spike.
- RPD Relative Percent Difference.
- NA Not applicable.

	% F	Recovery		RPD	
	Water	Soil/Sediment	Water	Soil/Sediment	
SVOC Surrogate Recovery Limits					
Compound					
Nitrobenzene-d5 (Base/Neutral)	56-112	38-105	NA	NA	
2-Fluorobiphenyl (Base/Neutral)	53-108	40-109	NA	NA	
Terphenyl-d14 (Base/Neutral)	50-122	16-151	NA	NA	
Phenol-d5 (Acid)	10-48	41-118	NA	NA	
2-Fluorophenol (Acid)	10-65	37-125	NA	NA	
2,4,6-Tribromophenol (Acid)	46-122	10-120	NA	NA	
LCS (Blank) and Matrix Spike Recovery and RPD	Limits				
Compound					
2,4,5-Trichlorophenol	67-114	50-115	30	30	
2,4,6-Trichlorophenol	67-111	53-118	30	30	
2,4-Dichlorophenol	64-107	58-115	30	30	
2,4-Dimethylphenol	55-100	65-112	30	30	
2,4-Dinitrophenol	19-113	10-129	30	30	
2,4-Dinitrotoluene	65-113	53-110	30	30	
2,6-Dinitrotoluene	68-114	51-115	30	30	
2-Chloronaphthalene	65-107	51-102	30	30	
2-Chlorophenol	53-101	56-110	30	30	
2-Methylnaphthalene	66-102	51-98	30	30	
2-Methylphenol	40-90	54-117	30	30	
2-Nitroaniline	73-116	51-109	30	30	
2-Nitrophenol	65-107	55-101	30	30	
3 & 4 Methylphenol	30-75	47-103	30	30	
3,3'-Dichlorobenzidine	69-129	24-105	30	30	
3-Nitroaniline	59-108	32-104	30	30	
1,6-Dinitro-2-methylphenol	58-115	10-110	30	30	
1-Bromophenyl phenyl ether	66-110	44-102	30	30	
4-Chloro-3-methylphenol	57-106	55-117	30	30	
4-Chloroaniline	58-105	10-96%	30	30	
4-Chlorophenyl phenyl ether	68-105	50-106	30	30	
4-Methylphenol	30-75	47-103	30	30	
4-Nitroaniline	49-119	45-106	30	30	
4-Nitrophenol	10-44	46-115	30	30	
Acenaphthene	66-108	46-100	30	30	

See footnotes on last page.

Table C-4B. SVOC Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	% F	Recovery		RPD	
	Water	Soil/Sediment	Water	Soil/Sediment	
LCS (Blank) and Matrix Spike Recovery and RPD L	imits				
Compound					
Acenaphthylene	67-107	51-103	30	30	
Acetophenone	68-109	40-95	30	30	
Anthracene	68-108	50-107	30	30	
Atrazine	56-116	30-100	30	30	
Benzaldehyde	52-150	10-160	30	30	
Benzo[a]anthracene	65-106	46-112	30	30	
Benzo[a]pyrene	58-101	36-89	30	30	
Benzo[b]fluoranthene	65-111	33-96	30	30	
Benzo[g,h,i]perylene	65-134	43-106	30	30	
Benzo[k]fluoranthene	66-114	35-115	30	30	
bis (2-chloroisopropyl) ether	65-107	45-102	30	30	
Bis(2-chloroethoxy)methane	69-108	51-100	30	30	
Bis(2-chloroethyl)ether	62-108	44-101	30	30	
Bis(2-ethylhexyl) phthalate	66-114	49-119	30	30	
Butyl benzyl phthalate	66-115	49-117	30	30	
Caprolactam	10-30	10-127	30	30	
Carbazole	67-110	49-104	30	30	
Chrysene	68-112	45-114	30	30	
Dibenz(a,h)anthracene	67-124	43-107	30	30	
Dibenzofuran	68-105	52-106	30	30	
Diethyl phthalate	66-109	52-114	30	30	
Dimethyl phthalate	69-111	52-112	30	30	
Di-n-butyl phthalate	68-111	50-108	30	30	
Di-n-octyl phthalate	51-115	40-106	30	30	
Diphenyl	66-112	50-105	30	30	
Fluoranthene	68-108	49-108	30	30	
Fluorene	68-105	51-108	30	30	
Hexachlorobenzene	65-107	43-104	30	30	
Hexachlorobutadiene	52-99	45-98	30	30	
Hexachlorocyclopentadiene	40-105	24-98	30	30	
Hexachloroethane	50-99	45-90	30	30	
Indeno[1,2,3-cd]pyrene	68-121	43-109	30	30	
Isophorone	68-108	46-97	30	30	
Naphthalene	63-101	53-94	30	30	
Nitrobenzene	66-106	42-106	30	30	

See footnotes on last page.

Table C-4B. SVOC Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	<u>%</u> R	% Recovery		RPD		
	Water	Soil/Sediment	Water	Soil/Sediment		
LCS (Blank) and Matrix Spike Recovery and RPD Limit	S					
Compound						
N-Nitrosodi-n-propylamine	70-109	42-107	30	30		
N-Nitrosodiphenylamine	71-121	49-106	30	30		
Pentachlorophenol	55-116	19-113	30	30		
Phenanthrene	68-110	48-108	30	30		
Phenol	12-44	54-115	30	30		
Pyrene	61-110	49-116	30	30		

See NYSDEC Revision 2005 for additional method performance criteria.

SVOC Semi-Volatile Organic Compound.

QC Quality Control.

LCS Laboratory Control Sample or Blank Spike.

RPD Relative Percent Difference.

NA Not applicable.

Table C-4C. Pesticide Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	% R	ecovery		RPD
	Water	Soil/Sediment	Water	Soil/Sediment
Pesticide Surrogate Recovery Limits				
Compound				
Tetrachloro-m-xylene	49-132	40-150	NA	NA
DCB Decachlorobiphenyl	37-144	53-150	NA	NA
CS (Blank) and Matrix Spike Recovery and RPD Limits				
Compound				
4,4'-DDD	68-136	63-150	30	30
4,4'-DDE	66-132	58-150	30	30
1,4'-DDT	66-132	57-150	30	30
Aldrin	61-122	58-143	30	30
Ipha-BHC	63-122	58-138	30	30
lpha-Chlordane	62-129	49-143	30	30
eta-BHC	64-119	60-139	30	30
lelta-BHC	62-124	60-141	30	30
Dieldrin	62-112	55-128	30	30
Endosulfan I	64-123	60-138	30	30
Endosulfan II	63-116	59-133	30	30
Endosulfan sulfate	56-121	56-133	30	30
Endrin	42-138	61-150	30	30
Endrin aldehyde	56-119	55-122	30	30
Endrin ketone	62-125	62-139	30	30
gamma-BHC (Lindane)	59-121	58-136	30	30
jamma-Chlordane	63-120	45-147	30	30
Heptachlor	61-118	58-137	30	30
Heptachlor epoxide	64-120	59-136	30	30
Methoxychlor	56-125	42-150	30	30
Toxaphene	70-130	70-130	30	30

See NYSDEC Revision 2005 for additional method performance criteria.

QC Quality Control.

LCS Laboratory Control Sample or Blank Spike.

- RPD Relative Percent Difference.
- NA Not applicable.

Table C-4D. PCB Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	% Recovery		RPD		
	Water	Soil/Sediment	Water	Soil/Sediment	
PCB Surrogate Recovery Limits					
Compound					
Decachlorobiphenyl	37-150	30-150	NA	NA	
LCS (Blank) and Matrix Spike Recovery and RPD Limits					
Compound					
Aroclor 1016	71-126	60-144	30	30	
Aroclor 1260	73-130	63-143	30	30	

See NYSDEC Revision 2005 for additional method performance criteria.

PCB Polychlorinated biphenyls

QC Quality Control.

LCS Laboratory Control Sample or Blank Spike.

RPD Relative Percent Difference.

NA Not applicable.

Table C-4E. Metals and Cyanide Analytical QC Limits, Quality Assurance Project Plan (QAPP), RI Work Plan, Former Dangman Park Manufactured Gas Plant Site, Brooklyn, New York.

	% R	ecovery		RPD
	Water	Soil/Sediment	Water	Soil/Sediment
QC Acceptance Criteria for Metals and Cyanide				
CRQL Check Standard				
TAL Metals (except mercury)	NA	NA	NA	NA
Fotal Cyanide	NA	NA	NA	NA
Free Cyanide	NA	NA	NA	NA
Continuing Calibration Verification				
TAL Metals (except mercury)	90-110	90-110	NA	NA
Mercury	80-120	80-120	NA	NA
Total Cyanide	90-110	90-110	NA	NA
Free Cyanide	NA	85-115	NA	NA
LCS				
TAL Metals (except mercury)	80-120	75-125	NA	NA
Aercury	85-115	75-125	NA	NA
Total Cyanide	90-110	90-110	NA	NA
Free Cyanide	NA	80-120	NA	NA
Laboratory Duplicate				
TAL Metals (except mercury)	NA	NA	20	20
Mercury	NA	NA	20	20
Total Cyanide	NA	NA	20	NA
Free Cyanide	NA	NA	20	35
Matrix Spike				
TAL Metals (except mercury)	75-125	75-125	NA	NA
Mercury	70-130	75-125	NA	NA
Total Cyanide	90-110	87-115	10	10
Free Cyanide	NA	75-125	NA	NA

Above control limits may not apply if the native sample concentration exceeds 4 times the spike concentration.

See NYSDEC Revision 2005 for additional method performance criteria.

QC Quality Control.

CRQL Contract Required Quantitation Limit.

LCS Laboratory Control Sample.

RPD Relative Percent Difference.

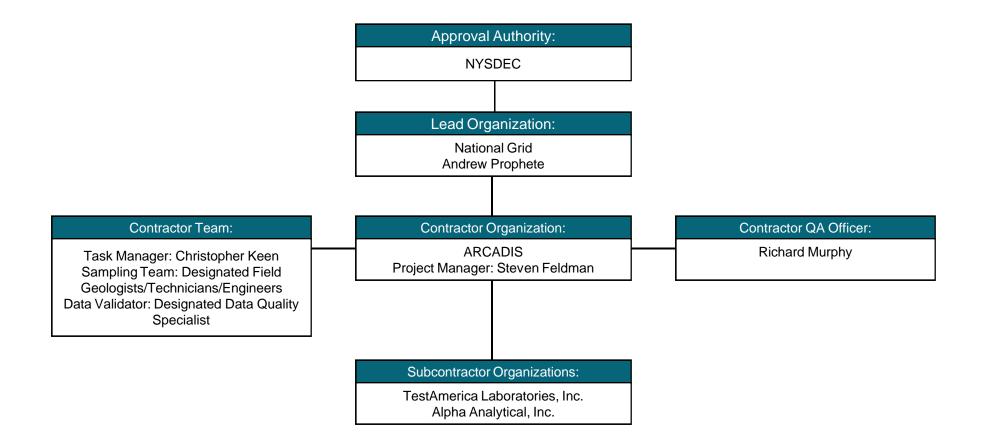
NA Not applicable.

#### Attachment C-1

Project Organizational Chart

#### **Project Organizational Chart**

Former Dangman Park MGP Site Brooklyn, NY



Attachment C-2

Field Forms

#### ARCADIS Sample/Core Log

Boring/Wel			Project Name/N	lo.			Page	of	
Site Location					Drilling Started	Drilling Completed			
Total Depth	Drilled		feet	Hole Diameter	inches	Type of Sample/ Coring Device			
Length and of Coring D						Sampling Interval		feet	
Land Surface	ce Elev.		feet	Surveyed	Estimated	Datum			
Drilling Met	hod					Drilling Fluid Used			
Drilling Contractor					Driller		Helper		
Prepared By					Hammer Weight	pounds	Hammer Drop	inches	;
Sample/Core (feet below la	and surface)	Recovery	Time/Hydraulic Pressure or Blows per 6						
From	То	(feet)	Inches	Sample/Core Des	scription				PID (ppm)

#### ARCADIS Sample/Core Log (Cont.d)

Boring/Wel	I			Page of	-
Prepared by	у				
Sample/Core (feet below la	e Depth and surface)	Core	Time/Hydraulic Pressure or Blows per 6		
From	То		Inches	Sample/Core Description	PID (ppm)
	Ī	1			



#### WELL CONSTRUCTION LOG

	Project Well
LAND SURFACE	Town/City
drilled hole	CountyState
	Permit No
✓ ——Well casing inch diameter	Land Surface and Measuring Point Elevation: Datum:
	Land Surfacefeet Surveyed
	Measuring Pointfeet Estimated
Grout	Installation Date(s)
	Drilling Method
	Drilling Contractor
	Drilling Fluid
ft*	Development Technique(s) and Date(s)
ft*	Fluid Loss During Driling gallons
ft*	Static Depth to Water feet below M.P.
Well Screen	Water Removed During Development gallons
inch diameter slot	Pumping Duration hours
	Well Purpose
Gravel Pack	Remarks
Formation Collapse	
ft*	
ft*	Prepared by
Measuring Point is Top of Well Casing Unless Otherwise Noted.	

\* Depth Below Land Surface

#### WELL DEVELOPMENT LOG

Project				Well			Date				
Developed	Ву:						Well	Material			
Initial Station	c Depth to V	Vater (ft bmp)					Well	Diameter			
Initial Total	Depth of W	/ell (ft bmp)					Total Rem	l Gallons oved			
Final Static Depth to Water (ft bmp)							Pump Decontaminated YES Prior to Development				
Final Total	Depth of W	ell (ft bmp)									
Time	Pumping	Depth	Gallons	Turbidity	Conductivity	pН		Temp		Appearance	

Time	Pumping Rate (gpm)	Depth to Water (ft bmp)	Gallons Removed	Turbidity (NTU)	Conductivity (mS/cm)	рН (s. u.)	Temp (ºC)	Appearance
		(						



Page \_\_\_\_\_ of \_\_\_\_\_

#### Low-Flow Groundwater Sampling Log

Project	<b></b>									
Project Number Date Sampling Time Weather			Site Location				Well ID			
			Sampled By							
			Recorded By							
				Coded Replica	ite No					
Instrument Ide	ntification									
						Serial #				
Water Quality I	vieter(S)	4								
Casing Materia	ıl			Purge	Method	_				
Casing Diamet	er			Pump Intake Depth (ft bmp)			Bottom  Finish			
Sounded Dept	h (ft bmp)									
Depth to Water	r (ft bmp)									
				Field Parameter	Measuremen	ts During Purging			<b>.</b>	
Time	Minutes Elasped	Flow Rate (mL/min)	Volume Purged	Temp (°C)	рН (s.u.)	Conductivity (umhos or mS/cm) <sup>1)</sup>	ORP (mV)	DO (mg/L)	Turbidity (NTU)	Depth to Water (ft bmp)
						,				
						++-				
Collected Sam	ple Condition		Color		Odor	<b>-</b>		Appearance_	-	
Parameter Container		Container			No.			Preservative		
	9	_			-			_		
		-			-				<u></u>	
		-	<u></u>		-			_		
PID Reading			_							
Comments										
				007777778788878787		014-15-29-16-16-1				

\*

#### Water Level Record

Project Name/No.

Page of

Date\_\_\_\_\_

Well(s)	Depth to Water (ft bmp)	Time	Remarks

Instrument Model Number Instrument Serial Number Calibration Gas ppm									
	[			Calibration					
Date/Time	Initials	Battery Check	Background Value	True Gas Value	Measured Gas Value	Adjust			
COMMENTS:									

### national**grid**

Ambient Air (Canister) Sample Collection Field Form

Project # Project Name		Consultant			
Sample ID		Vacuum gauge "zero" ("Hg) Start Pressure ("Hg)			
End Date/Time		End Pressure ("Hg) End pressure > "zero"? Sampling duration (intended)			
Canister ID					
Flow controller ID					
Tubing type used	Length of tubing	cm Tubing volume	cc		
Volume purged	cc @min	1 to 3 volumes purged @ < 200cc/min?			
Weather Conditions at Start of San	pling:				
Air temperature (°F)	Rainfall	Wind direction			
Barometric pressure	Relative humidity	Wind speed (mph)			
Substantial changes in weather cor	ditions during sampling or over the pa	ast 24 to 48 hrs:			

Site Plan showing sample location, building(s) being sampled, building HVAC inlet, outdoor air sources, wind direction

Comments:

### national**grid**

Soil Vapor (Canister) Sample Collection Field Form

Project #		Consultant Collector			
Sample ID		Vacuum gauge "zero" ("Hg)			
Start Date/Time		Start Pressure ("Hg)			
End Date/Time		End Pressure ("Hg)			
Canister ID		End pressure > "zero"?			
Flow controller ID		Sampling duration (intended)			
Associated ambient air sample ID		Depth of sample point below grade			
Tubing type used	Length of tubing	cm Tubing volumecc			
Volume purged	_cc @	min 1 to 3 volumes purged @ < 200cc/min?			
Chamber tracer gas conc.		Tracer gas conc. during purging			
Weather Conditions during Probe Insta	allation:				
Air temperature (°F)	Rainfall	Wind direction			
Barometric pressure	_	Wind speed (mph)			
Substantial changes in weather condit	ions during sampling or ove	er the past 24 to 48 hrs:			
Weather Conditions at Start of Sampli	ng:				
Air temperature (°F)	Rainfall	Wind direction			
Barometric pressure		Wind speed (mph)			
Substantial changes in weather condit	ions during sampling or ove	er the past 24 to 48 hrs:			
Site Plan showing sample location, bu	uildings, landmarks, potenti	al soil vapor and outdoor air sources, preferential pathways			
Comments:					

# **ARCADIS**

# Attachment C-3

Chain-of-Custody Form

#### **CHAIN OF CUSTODY & LABORATORY** ANALYSIS REQUEST FORM Page \_\_\_\_ of \_\_\_\_

Lab Work Order #

						1					1					
Contact & Company Name:	Telephone:					Preservative								Preservation Ke	Keys ev: Contain	er Information Key:
Address: City State Zip	-		Filtered (✓)								A. H <sub>2</sub> SO <sub>4</sub> B. HCL	1. 40 m	l Vial			
Address:	Fax:					# of Container	s							B. HCL C. HNO <sub>3</sub>	2. 1 L A 3. 250 r	
Re						Container Information								D. NaOH	<ol> <li>4. 500 r</li> <li>5. Enco</li> </ol>	
City State Zip	E-mail Addre	SS:					PAF	RAMETE		LYSIS 8	METH	OD		F. Other:	6. 2 oz.	Glass
ō							/	/	/	/	/	/	/	E. None F. Other: G. Other:	7. 4 oz. 8. 8 oz.	
Project Name/Location (City, State):	Project #:					4 /								H. Other:	9. Othe	
															10. Othe	r:
Sampler's Printed Name:	Sampler's Si	gnature:												Matrix Key: SO - Soil	SE - Sediment	
			_			1 /								W - Water T - Tissue	SL - Sludge A - Air	SW - Sample Wipe Other:
Sample ID	Colle	ection	Туре	€(√)	Matrix		/				/					ounor
•	Date	Time	Comp	Grab			/ ,	/ ,	/			/	/	REMARKS	5	
Special Instructions/Comments:								l	Special Q	A/QC Instruc	tions(√):					
Laboratory Information	1				Dista		luished By			Received By			elinquished		Laboratory F	Received By
Lab Name:	Cooler C	ustody Se	ai (✓ )		Printed	I Name:			Printed Name:			Printed Name:		Prin	ited Name:	
	Inta	act		ot Intact	Signati	ure:			Signature:			Signature:		Sin	nature:	
$\Box$ Cooler packed with ice ( $\checkmark$ )				n intact	Gigridu				organitation of			Signature.			intero.	
Specify Turnaround Requirements:	Sample I	Receipt:			Firm:				Firm/Courier:			Firm/Courier:		Firm	n:	
	Campier	tooopt.														
Shipping Tracking #:	Condition	n/Cooler Te	emp:		Date/T	ime:			Date/Time:			Date/Time:		Dat	e/Time:	
20730826 CofC AR Form 01.12.2007		Dis	tribution	:	WHITE -	- Laboratory	returns w	ith results		`	YELLOW -	Lab copy		P	NK – Retained I	ov ARCADIS

# **ARCADIS**

# Attachment C-4

TestAmerica Laboratories, Inc. Quality Assurance Manual and Standard Operating Procedures (SOPs)



# **Cover Page:**

# **Quality Assurance Manual**

TestAmerica Edison 777 New Durham Road Edison, NJ 08817 732-549-3900 732-549-3679

www.testamericainc.com

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# Title Page:

# Quality Assurance Manual Approval Signatures

Drive gladwell

Laboratory Director - Ann Gladwell

Quality Manager - Carl Armbruster

**Operations Manager - Mark Acierno** 

Date: 9/25/09

Date: 9/25/09

Date: 9/25/09

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# **REFERENCED CORPORATE SOPs AND POLICIES**

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

# **REFERENCED LABORATORY SOPs**

SOP Reference	Title
ED-GEN-001	Data Management and Handling Procedures
ED-GEN-002	Document Control
ED-GEN-003	Control of Non-Conformances and Corrective Action
ED-GEN-007	Subsampling
ED-GEN-011	Calibration and Use of Laboratory Pipettes
ED-GEN-014	Thermometer Calibration
ED-GEN-021	Data Review
ED-GEN-022	Training
ED-GEN-024	Record Storage and Retention
ED-RP-001	Reports Production
ED-SPM-001	Sample Receipt, Login, Identification and Storage
ED-SPM-006	Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soil
ED-SPM-007	Disposal of Samples and Associated Laboratory Waste

#### SECTION 3

#### INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Edison's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, EPA, Revised July 1991.
- EPA 600/R-95/131, Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3<sup>rd</sup> Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration.* Document ILM04.0.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration*. Document Number OLMO3.1, August 1994.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.

#### 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization. Refer to Appendix 2 for the Glossary/Acronyms.

# 3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical and physical parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in TestAmerica Edison Work Instruction EDS-WI-009 (Edison Analytical Capabilities). The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

# 3.4 MANAGEMENT OF THE MANUAL

#### 3.4.1 <u>Review Process</u>

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. ED-GEN-002).

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#### SECTION 4

#### ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 <u>OVERVIEW</u>

TestAmerica Edison is a local operating unit of TestAmerica Laboratories, Inc.The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. TestAmerica Edison has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The TestAmerica Edison laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Edison is presented in Figure 4-1.

#### 4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### 4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Edison laboratory.

#### 4.2.2 Laboratory Director/Lead Technical Director

TestAmerica Edison's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to the General Manager (GM). The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Serves as lead technical director for all fields of testing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.

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- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Monitors standards of performance in quality control and quality assurance.
- Monitors the validity of analyses performed and data generated in the lab to assure reliable data.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Interfaces with Project Management and Customer Service to forecast receipts, provide quality analytical data to clients and meet on-time delivery dates.
- Ensures that the facility has appropriate Information Technology resources and that they are used effectively to support operational requirements.
- Actively participates in the process of sharing and adopting best practices within TestAmerica. Provides technical assistance to other TestAmerica laboratories as needed to improve productivity and customer service.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, the Operations Manager, the Project Management Director, the Client Services Manager, the Service Center Manager, the Environmental, Health and Safety Manager and the Support Services Manager as direct reports.

### 4.2.3 <u>Quality Assurance (QA) Manager</u>

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA staff to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.

- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for and conducting the annual internal audits of quality systems and lab technical operations.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Monitoring standards of performance in quality control and quality assurance.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review and approval of MDL studies.
- Review and approval of analyst Demonstrations of Capability (IDOC/CDOC).
- Review and approval of statistical control limit evaluations.
- Maintenance of quality reference limits in LIMS (TALS).
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

## 4.2.4 Quality Assurance (QA) Specialist

The Quality Assurance (QA) Specialist is responsible for performing data audits, special audits, assisting with external and systems audits, overseeing the maintenance of QC records, certifications, Standard Operating Procedures (SOPs), training records, DOCs, arranging and managing PT samples. Additional responsibilities may include assisting with systematic problems within the laboratory, assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts and other functions in support of the QA Manager's responsibilities as assigned.

• Assist QA Manager in conducting QA training courses, including ethics training.

- Performs data audits.
- Assist in performing special audits as deemed necessary by data audits, client inquiries, etc.
- Assisting in, conducting and responding to external audits conducted by clients and regulatory agencies.
- Assisting in reviewing and/or writing of Quality Assurance Project Plans, and technical and QC specifications in contracts.
- Maintaining all necessary laboratory certifications.
- Arranging and managing PT samples.
- Reviewing laboratory SOPs. Writing SOPs as needed.
- Maintaining historical indices of all technical records including SOPs, QC records, laboratory data, etc.
- Ensuring maintenance of records archives.
- Assisting in and monitoring laboratory's method compliance.
- Ensuring maintenance of DOCs for all analysts.
- Ensuring maintenance of training records for all employees.
- Assisting in identification of systematic problems within laboratories.
- Recommends resolutions for ongoing or recurring nonconformance.
- Providing statistical feedback to departments on error rates, and assisting in identifying systematic improvements to minimize errors.
- Assists in tracking of customer complaints, providing statistical feedback to the laboratory, and assisting in identifying improvements.
- Overseeing and reviewing MDL studies.
- Ensuring control charts are generated; oversees and approves setting of control limits.
- Assists in monitoring new regulations and communicating them to the laboratory.

# 4.2.5 LAN Analyst

The LAN Analyst reports directly to the Regional Desktop Support Supervisor. Responsibilities include:

- Works with Corporate IT to solve information systems problems and to standardize laboratory IT equipment and processes.
- Monitors and supports office automation so that LAN is operational for internal and external communications.
- Troubleshoots problems throughout laboratory relating to computers, software, telephones and other electronic equipment.
- Responsible for new user setup on network, LIMS, telephone and voice mail.

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- Installs or upgrades computers and other equipment.
- Maintains tape backups for multiple computer servers including LIMS.
- Maintains historical files of software, software operating procedures (manuals), software changes/modifications (Change Log) and software version numbers.
- Maintains log of repairs and service performed on LIMS hardware.
- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.

#### 4.2.6 **Operations Manager**

The Operations Manager manages and directs the analytical and reports production sections of the laboratory. He/She reports directly to the Laboratory Director. Specific responsibilities include:

- Maintains awareness of any environmental conditions of the facility housing the LIMS that may compromise LIMS raw data and informs management.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Laboratory Director and QA Manager and in compliance with regulatory requirements.
- Works with the Department Managers to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

#### 4.2.7 Environmental, Health and Safety Manager

The Environmental, Health and Safety Manager reports directly to the Laboratory Director. The duties of this position consist of:

- Supervises the Environmental, Health and Safety/Facilities Team.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.

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- Oversee the inspection and maintenance of general safety equipment fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.
- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

#### 4.2.8 EH&S/Facilities Coordinator

The EH&S/Facilities Coordinator reports directly to the Environmental, Health and Safety Manager. The duties of this position consist of:

- Monitors laboratory for unsafe conditions or acts to keep lab in compliance with the Chemical Hygiene Plan, EH&S Procedures, and company policies.
- Ensures the proper personal protective equipment is available and personnel are properly trained in its use.
- Assists the Environmental, Health and Safety Manager in the investigation of accidents, incidents, and near misses and identifies and eliminates root cause.
- Conducts monthly facility inspections for compliance with health, safety and environmental regulations and procedures. Completes and forwards monthly inspection report to safety committee and laboratory management for corrective actions.
- Conducts safety equipment checks to ensure proper working order and sufficient inventory.
- Plans and tracks completion of monthly general awareness training sessions and compliance training, including new employee EH&S orientation.
- Coordinates emergency response team to provide prompt medical attention and stabilize emergency situation. After emergency is over, assists in determining appropriate clean up procedures.

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- Conducts the monthly EH&S committee meeting.
- Participates in monthly EH&S conference call.
- Reviews and maintains MSDS's for laboratory materials.
- Coordinates the management and disposal of laboratory wastes.
- Assists in the preparation and maintenance of the laboratory Integrated Contingency Plan.
- Monitors air quality in facility, including monitoring fumehoods for proper operation and ventilation.
- Maintains overall building facilities and equipment as well as administers prevention maintenance measures.
- Contacts outside contractors as necessary to repair/maintain items outside the realm of reasonable maintenance.
- Performs miscellaneous errands, buying parts for labs, janitorial supplies.
- Oversees storage facilities, files and outside storage.

#### 4.2.9 Department Managers

Department Managers report to the Operations Manager and typically serve as the Technical Director of their respective departments. Responsibilities include:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- Participates in the selection, training (including familiarization with SOP, QC, Safety, and computer systems), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts. Ensure the documentation of these activities in accordance with systems developed by the QA and Personnel Departments.
- Provide technical guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Operations Manager, and/or QA Manager.
- Ensures that 100% of data review undergoes two documented levels of review. Likewise ensures that all non-conformance issues are properly documented.
- Responsible for the timely and accurate completion of performance evaluation samples and MDLs, for the department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.

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- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Provide written responses to external and internal audit issues.

## 4.2.10 Laboratory Analysts and Technicians

Laboratory analysts and technicians are responsible for conducting analysis and performing all tasks assigned to them by their department manager or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database by means of Non-Conformance Memos (NCMs).
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their Department Manager, the Laboratory Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated and document the review in the raw data and on the review checklist prior to entering and submitting for secondary level review.
- Suggest method improvements to the Department Manager, the Laboratory Director, and the QA Manager. These improvements, if approved, will be incorporated within the constraints of the consensus reference methods.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
- Adhere to all environmental, health and safety protocols and attend safety meetings as required.
- Attend and participate in all staff meetings.

# 4.2.11 Sample Control Manager

The Sample Control Manager reports to the Laboratory Director. The responsibilities are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Ensure the verification of data entry from login.
- Manages the preparation and shipment of bottle kits to clients.
- Oversees the responsibilities of all Sample Control Technicians.
- Supervises the storage and disposal of all samples.

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### 4.2.12 Customer Service Manager

The Customer Service Manager reports to the Laboratory Director and serves as the primary interface between the laboratory and the Sales and Marketing staff. Responsibilities include:

- Laboratory's primary client representative.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Compiles and interprets receipts forecast to show near term business trends.
- Manages a minimal list of projects/programs for key client accounts. (Note: sufficient time is needed to manage the PM group and the CSM must not be overwhelmed with project management.)
- Prepares proposals for new business opportunities.
- Compiles and interprets Bid Activity Report.
- Compiles and interprets receipts forecast to show near term business trends.
- Prepares proposals for new business opportunities.
- Provides general sales support to Account Executives for business development activities started in the field.
- Develops and maintains business materials and organized information resource files that include project descriptions, resumes, original proposals, boilerplates, and company qualifications materials.

#### 4.2.13 Director of Project Management

The Director of Project Management reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible for ensuring that clients receive the proper sampling supplies, as appropriate.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and guality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.

- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

## 4.2.14 Project Manager

The Project Managers report directly to the Director of Project Management and serve as liaisons between the laboratory and its clients. The Project Manager's responsibilities include:

- Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory.
- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Monitor the status of all projects in-house to ensure timely delivery of reports.
- Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Coordinate client requests for sample containers and other services.
- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinate subcontract work.
- Respond to client inquiries concerning sample status.
- Performs final completeness review of data packages prior to release to client.

# 4.2.15 Project Management Assistant

The Project Management Assistant coordinates and monitors scheduling, timely completion and maintenance of project documentation files and completion of project set up and final report review, invoicing, and EDD's. Assists the Project Manager in servicing the client's needs. Specific responsibilities include:

- Reviews login confirmation reports for accuracy and corrects as needed.
- Generates diskettes for electronic data deliverables (EDD's) for electronic delivery to clients.

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- Enters data that was subcontracted to other laboratories.
- Monitors report due dates for timely delivery.
- Assists Project Manager in changing compound lists, TAT, deliverables and other client specific requirements in the LIMs project and/or job database.
- Invoices completed data packages and generates credit or debit invoices to ensure proper payment.

#### 4.2.16 Service Center Manager

The Service Center Manager (SCM) manages the service center and acts as a liaison between the laboratory and the local client base. The SCM is in charge of maintaining the Service Center facility, managing service center couriers, samplers and other personnel, and working with sales to develop, maintain and grow the client base in the area.

- Local area primary client representative for service center location.
- May head project start up meetings to ensure project objectives are successfully met and hands off project detail to assigned Project Manager(s).
- Works with the Quality Assurance Manager and Account Executives (AE) to evaluate and establish project requirements for the service center area.
- Ensures client complaints are handled professionally, and resolved in a timely manner.
- Is in charge of scheduling service center couriers and samplers, preparing bottle orders for delivery, scheduling sample pick ups and shipping samples to the designated laboratory for analysis.
- May manage a minimal list of projects/programs for key client accounts.
- Maintains the facilities at the service center and is responsible for all EH&S policies of TestAmerica at the service center.
- Responsible for all company vehicles that operate out of the service center.
- Provides general sales support to AEs for business development activities started in the field.
- Prepares proposals for new business opportunities.
- Orders supplies (bottles, coolers, etc.) for the service center

#### 4.3 DEPUTIES

The following table defines who assumes the responsibilities of key personnel in their absence:

Key Personnel	Deputy
Laboratory Director	In the event of absence the Laboratory Director's responsibilities are shared by the Laboratory Operations Manager, the Quality Assurance Manager and the Client Services Manager, as appropriate.
Laboratory Operations Manager	Laboratory Director

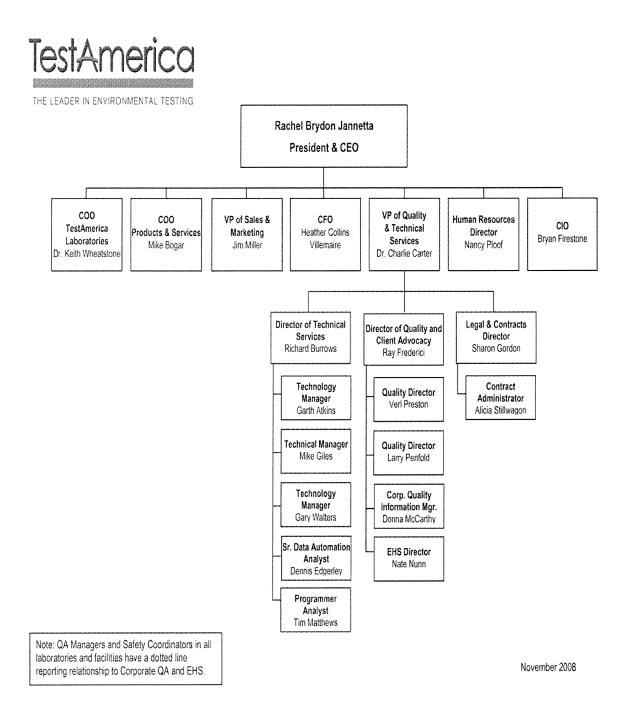
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Key Personnel	Deputy
QA Manager	Laboratory Director
_	QA Specialist
Analytical Department Managers	Operations Manager
Client Services Manager/Director of Project	Laboratory Director
Management	
EH&S Manager	EH&S Coordinator
Sample Control Manager	Sample Control Supervisor
Service Center Manager	Field Services Supervisor

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Figure 4-1.

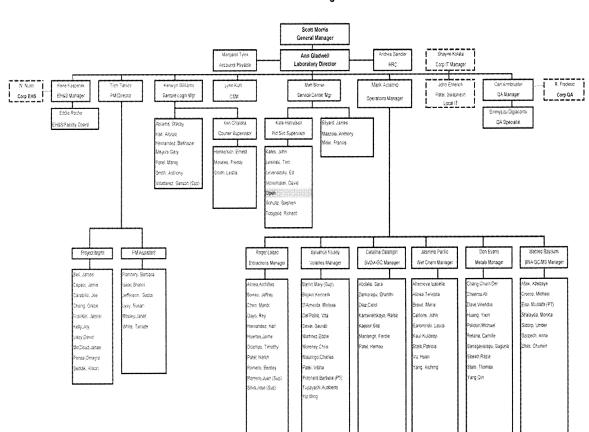
# **Corporate and Laboratory Organization Charts**



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# Figure 4-1. (continued)

## **Corporate and Laboratory Organization Charts**



#### TestAmerica Edison Organization

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#### **SECTION 5**

#### QUALITY SYSTEM (NELAC 5.4.2)

#### 5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

# 5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001.)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 16).
- Produce results, which are accurate and include QA/QC information that meets client predefined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

# 5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual Each laboratory has a lab specific quality assurance manual.
- <u>Corporate SOPs and Policies</u> Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- <u>Work Instructions</u> A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- <u>Laboratory SOPs</u> General and Technical
- <u>Corporate Quality Policy Memorandums</u>

#### 5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases.

## 5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

#### 5.4.1 <u>Precision</u>

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

#### 5.4.2 <u>Accuracy</u>

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

#### 5.4.3 <u>Representativeness</u>

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the

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procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

## 5.4.4 <u>Comparability</u>

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

#### 5.4.5 <u>Completeness</u>

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

#### 5.4.6 <u>Selectivity</u>

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

# 5.4.7 <u>Sensitivity</u>

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

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### 5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains Quality Control Limit tables within TALS (the laboratory's LIMS) that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 25.

## 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 25. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

# 5.6.1 <u>QC Charts</u>

The QA Manager evaluates these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file.

#### 5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 17). These metrics are used to drive continuous improvement in the laboratory's Quality System.

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### **SECTION 6**

#### DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 <u>OVERVIEW</u>

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. ED-GEN-002 (Document Control).

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and Corrective Action Reports (CARS). Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

#### 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are

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identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year and revised as appropriate. Changes to documents occur when a procedural change warrants.

#### 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. ED-GEN-002 (Document Control). Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder and on the Edison intranet (EdiNET).

For changes to SOPs, refer to SOP No. CW-Q-S-002, Writing a Standard Operating Procedure SOP. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. A master list of work instructions is maintained by the QA department and electronic versions are kept on the network drive. The procedure for the care of these documents is in SOP ED-GEN-002 (Document Control).

#### 6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. ED-GEN-002 (Document Control).

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#### **SECTION 7**

#### SERVICE TO THE CLIENT (NELAC 5.4.7)

### 7.1 <u>OVERVIEW</u>

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

# 7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below).

- Legal & Contracts Director
- General Manager
- The Laboratory Project Management Director
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements. The Legal & Contracts Director maintains copies of all signed contracts. *The applicable Project Manager maintains local copies of signed contracts.* 

# 7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. These records are maintained in the project file by the Project Manager and/or Key Account Executive.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Lab Director.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

# 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

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The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

# 7.4 <u>SPECIAL SERVICES</u>

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 16 and 26).

**Note:** ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 26. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

# 7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors are available to discuss any technical questions or concerns that the client may have.

# 7.6 <u>REPORTING</u>

The laboratory works with our clients to produce any special communication reports required by the contract.

#### 7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

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# **SECTION 8**

# SUBCONTRACTING OF TESTS (NELAC 5.4.5)

# 8.1 <u>OVERVIEW</u>

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

# 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE) or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable, (e.g., on the

subcontractors NELAC, A2LA accreditation or State Certification).

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will

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notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

# 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

**Note:** The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

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# 8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

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Yes No

Yes No

Yes\_\_\_\_\_No\_\_\_\_\_

#### Figure 8-1.

#### Example - Subcontracted Sample Form

#### Date/Time:

### Subcontracted Laboratory Information:

- Subcontractor's Name:
- Subcontractor Point of Contact:
- Subcontractor's Address:
- Subcontractor's Phone:
- Analyte/Method:
- Certified for State of Origin:
- NELAC Certified:
- A2LA (or ISO 17025) Certified:
- CLP-like Required: (Full doc required)
- Requested Sample Due Date: (Must be put on COC)

#### **Project Manager:**

Laboratory Sample # Range: (Only of Subcontracted Samples)

Laboratory Project Number (Billing Control #):

All subcontracted samples are to be sent via bonded carrier and Priority Overnight. Please attach tracking number below and maintain these records in the project files.

Ρ	М	Si	qn	ati	ure	Э

Date

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### **SECTION 9**

### PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

### 9.1 <u>OVERVIEW</u>

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

# 9.2 <u>GLASSWARE</u>

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

# 9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pretested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

#### 9.3.1 <u>Purchasing</u>

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. The analyst may check the item out of the on-site consignment system that contains items approved for laboratory use.

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If an item is not available from the on-site consignment, the analyst must provide the master item number (from the master item list that has been approved by the Operations Manager), item description, package size, catalogue page number, and the quantity needed. If an item being ordered is not the exact item requested, approval must be obtained from the Operations Manager prior to placing the order. The Department Manager or the Laboratory Operations Manager places the order.

# 9.3.2 <u>Receiving</u>

It is the responsibility of the Facilities Coordinator to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

# 9.3.3 <u>Specifications</u>

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained

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Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

# 9.3.4 <u>Storage</u>

Reagent and chemical storage is important from the aspects of both integrity and safety. Lightsensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

# 9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Laboratory Operations Manager and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the

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requirements. The appropriate written requests are completed and the Laboratory Operations Manager places the order.

Upon receipt of a new or used piece of equipment, an equipment asset tag is affixed and the equipment is assigned a unique instrument ID ('BNAMS12', for example) that will be used to identify the instrument in LIMS and in logbooks. The instrument/equipment ID number is provided to the QA department which maintains the master laboratory equipment list. The IT department is also be notified so that the instrument can be added to the routine data back-up schedule. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 20). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the IT Department. The manufacturer's operation manual is retained at the bench.

# 9.5 SERVICES

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 21. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Laboratory Director and/or the Laboratory Operations Manager.

# 9.6 <u>SUPPLIERS</u>

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

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As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

# 9.6.1 <u>New Vendor Procedure</u>

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technology Director are consulted with vendor and product selection that have an impact on quality.

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# **SECTION 10**

#### <RESERVED>

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### **SECTION 11**

### COMPLAINTS (NELAC 5.4.8)

#### 11.1 <u>OVERVIEW</u>

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 13 (Corrective Actions) and is documented following the procedures in TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action).

#### 11.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to TestAmerica Edison SOP No. ED-GEN-003 (Control of Non-Conformances and Corrective Action.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

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The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

# 11.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 13.

# 11.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 17).

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# SECTION 12

# CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

# 12.1 <u>OVERVIEW</u>

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Department Manager for resolution. The manager may elect to discuss it with the Lab Director and/or QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 13. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 20. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Lab Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

# 12.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CA-L-S-001) outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, the Lab Operations Manager, a Department Manager, or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to

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reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised\_of the Laboratory Director, Laboratory Operations Manager, the QA Manager, and the Department Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures <u>must</u> be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, Laboratory Operations Manager, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

# 12.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

# 12.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

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### 12.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 12.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 13 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Laboratory Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

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# **SECTION 13**

# CORRECTIVE ACTION (NELAC 5.4.10)

# 13.1 <u>OVERVIEW</u>

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Data Inquiry, Client Complaint and Corrective Action Report Form (CAR) (TestAmerica Edison Work Instruction No. EDS-WI-012) (refer to Figure 13-1).

# 13.2 <u>GENERAL</u>

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track client complaints and provide resolution.

**13.2.1** <u>Data Inquiry/Client Complaint</u> – The CAR form is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints

**13.2.2** <u>Corrective Action Report (CAR)</u> – The CAR form is also used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCRs.
- Issues found while reviewing NCRs that warrant further investigation.
- Internal and external audit findings
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors

# 13.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

# 13.3.1 <u>Cause Analysis</u>

- Upon discovery of a non-conformance event, the event must be defined and documented. A CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Laboratory Director, Laboratory Operations Manager, or QA Manager (or QA designee) is consulted.

# 13.3.2 <u>Selection and Implementation of Corrective Actions</u>

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR is used for this documentation.

# 13.3.3 Monitoring of the Corrective Actions

- The Department Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each CAR is entered into a database for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly CARs for trends. Highlights are included in the QA monthly report (refer to Section 17). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the outof-control situation and problems encountered in solving the situation.

# 13.3.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as
  possible when the identification of a nonconformance casts doubt on the laboratory's
  compliance with its own policies and procedures, or on its compliance with state or federal
  requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 16.2.4, Special Audits.)

# 13.4 <u>TECHNICAL CORRECTIVE ACTIONS</u>

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 12). The documentation of these procedures is through the use of a CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The QA Department also maintains various Work Instructions detailing lab specific technical criteria (ex., laboratory generated QC limits).

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 20 and 21. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified and appropriate corrective action (e.g., reanalysis) is taken and documented.

# 13.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated. When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

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# Figure 13-1. Corrective Action Report

afe	Request Form / Corre	Job #:	Send Resp Name:	
			Address:	
afe				
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ontact:		Bound Reduced	Email	
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	Sample/Analysis	Results in Question Insul	îcient Data for V	alidationEDD
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Missing I	Pages	Calibration in Question		
Explanation	of Details:			
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Required Ac		a a suive dia terra de la contra		Actions Completed:
√ if needed	Department	Actions Required:		Initials: Date:
	PM			
	LOGIN			
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Final Appro	val of Data Inquiry Actions	Taken:		
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Int	iator Signature:	Date:		
LAB ERRO	YES NO (IF YES, PL	EASE COMPLETE SECTIONS 5 - 7) CORRECTIVE	ACTION ID#:	
Quality Asso	rance Review and Assignm	ent of Further Action: (to be completed by (24 Manager - uso page	2 if needed)	
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Super	tion of Corrective Action: <i>a</i> visor Signature: trance Final Approval <i>(24 1</i> )		Date:	
Super	visor Signature:		Date:	

# Table 13-1.

# **General Corrective Action Procedures**

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank <i>(Analyst)</i>	- Instrument response < MDL.	<ul> <li>Prepare another blank.</li> <li>If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc</li> </ul>
Initial Calibration Standards (Analyst, Supervisor)	<ul> <li>Correlation coefficient &gt; 0.99 or standard concentration value.</li> <li>% Recovery within acceptance range.</li> <li>See details in Method SOP.</li> </ul>	<ul> <li>Reanalyze standards.</li> <li>If still unacceptable, remake standards and recalibrate instrument.</li> </ul>
Independent Calibration Verification (Second Source) (Analyst, Supervisor)	- % Recovery within control limits.	<ul> <li>Remake and reanalyze standard.</li> <li>If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.</li> </ul>
Continuing Calibration Standards (Analyst, Data Reviewer)	% Recovery within control limits.	<ul> <li>Reanalyze standard.</li> <li>If still unacceptable, then recalibrate and rerun affected samples.</li> </ul>
Matrix Spike / Matrix Spike Duplicate (MS/MSD) (Analyst, Data Reviewer)	- % Recovery within limits documented in TALS and/or Work Instructions	<ul> <li>If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS.</li> <li>If the LCS is within acceptable limits the batch is acceptable.</li> <li>The results of the duplicates, matrix spikes and the LCS are reported with the data set.</li> </ul>
Laboratory Control Sample (LCS) (Analyst, Data Reviewer)	- % Recovery within limits specified in TALS and/or Work Instructions	<ul> <li>Batch must be re-prepared and re- analyzed.</li> <li>Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags.</li> </ul>
Surrogates (Analyst, Data Reviewer)	- % Recovery within limits of method or within three standard deviations of the historical mean.	- Individual sample must be repeated. Place comment in LIMS.
Method Blank (MB_ (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	<ul> <li>Reanalyze blank.</li> <li>If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.</li> </ul>

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Proficiency Testing (PT) Samples (QA Manager, Department Manager/Supervisor)	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Internal / External Audits (QA Manager, Department Manager/Supervisor, Laboratory Director/Manager)	- Defined in Quality System documentation such as SOPs, QAM, etc	- Non-conformances must be investigated through CAR system and necessary corrections must be made.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager/ Supervisor, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001 or the Corrective Action SOP (ED-GEN-003).
Client Complaints (Project Managers, Lab Director/Manager, Sales and Marketing)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow- up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director/Manager, Department Supervisors/Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.

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QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Health and Safety Violation (Safety Officer, Lab Director/Manager, Department Supervisor/Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through CAR system.

#### Note:

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, toluene, acetone, 2-butanone and phthalates **provided** they appear in similar levels in the reagent blank and samples. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit

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### SECTION 14

### PREVENTIVE ACTION (NELAC 5.4.11)

### 14.1 <u>OVERVIEW</u>

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**14.1.1** The following elements are part of a preventive action system:

- <u>Identification</u> of an opportunity for preventive action.
- <u>Process</u> for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- Execution of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- <u>Close-Out</u> by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

**14.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 16). A highly detailed recap is not required; a simple

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recount of success and failure within the preventive action program will provide management a measure for evaluation.

# 14.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these various tracking indicators, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

- SOP Tracking Current Revisions w/ Effective Dates Required Biennial Revisions w/ Due Date
- Proficiency Testing (PT) Sample Tracking Pass / Fail – most current 2 out of 3 studies.
- Instrument / Equipment List
   Current / Location
- Accreditations New / Expiring
- Method Capabilities Current Listing by program (e.g., Potable Water, Soils, etc.)
- Key Personnel
   Technical Managers, Department Supervisors, etc..

These items are maintained on TestAmerica's Intranet (Proposal Library) or on our internal database (TotalAccess) which uploads to our company internet site.

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### **SECTION 15**

### CONTROL OF RECORDS (NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

# 15.1 <u>OVERVIEW</u>

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 15-1. Quality records (QA records) are maintained by the QA department and are indexed in a database, which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by Laboratory Operations under the direction of the Laboratory Operations Manager.

	Record Types <sup>1</sup> :	Retention Time:
Technical Records	- Raw Data - Logbooks <sup>2</sup> - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	<ul> <li>Quality Assurance Manual (QAM)</li> <li>Work Instructions</li> <li>Policies</li> <li>SOPs</li> <li>Manuals</li> </ul>	5 Years from document retirement date*
QA Records	<ul> <li>Internal &amp; External Audits/Responses</li> <li>Certifications</li> <li>Corrective/Preventive Actions</li> <li>Management Reviews</li> <li>Method &amp; Software Validation / Verification Data</li> <li>Data Investigation</li> </ul>	5 Years from archival* <u>Data Investigation:</u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	<ul> <li>Sample Receipt &amp; COC</li> <li>Documentation</li> <li>Contracts and Amendments</li> <li>Correspondence</li> <li>QAPP</li> <li>SAP</li> <li>Telephone Logbooks</li> <li>Lab Reports</li> </ul>	5 Years from analytical report issue*

#### Table 15-1. Record Index<sup>1</sup>

	Record Types <sup>1</sup> :	Retention Time:
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

<sup>1</sup> Record Types encompass hardcopy and electronic records.

- <sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).
- \* Exceptions listed in Table 15-2.

All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or a secure offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 15-2 have lengthier retention requirements and are subject to the requirements in Section 15.1.3.

# 15.1.1 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 15-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
NY Potable Water NYCRR Part 55-2	10 years

# Table 15-2. Example: Special Record Retention Requirements

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**15.1.2** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).

**15.1.3** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored in the laboratory's hard copy project file (in addition to the scanned copy included in the analytical report PDF). The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept in the project file as well. For additional details please refer to refer to TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Reference TestAmerica Edison SOP No. ED-GEN-024 (Record Storage and Retention).
- Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.

- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning
  process can be verified in order to ensure that no data is lost and the data files and storage
  media must be tested to verify the laboratory's ability to retrieve the information prior to the
  destruction of the hard copy that was scanned.
- Also refer to Section 20.14.1 'Computer and Electronic Data Related Requirements'.

# 15.2 TECHNICAL AND ANALYTICAL RECORDS

**15.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing results.

**15.2.2** Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**15.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;

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- sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

# 15.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

# 15.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms;

and

• procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

# 15.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 15-1.

# 15.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

**15.5.1** All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

**15.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

**15.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

**15.5.4** The laboratory has a records management system for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Records are considered archived when noted as such in the records management system.

# 15.5.5 <u>Transfer of Ownership</u>

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

# 15.5.6 <u>Records Disposal</u>

**15.5.6.1** Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 15-1 and 15-2).

15.5.6.2 Electronic copies of records must be destroyed by erasure or physically damaging

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off-line storage media so no records can be read.

**15.5.6.3** If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

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### **SECTION 16**

## AUDITS (NELAC 5.4.13)

## 16.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director	<ul> <li>All SOPs within a 2-year period</li> <li>All new analysts or new analyst/methods within 3 months of IDOC</li> </ul>
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

Table 16-1.	Types of Internal	Audits and Frequency
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## 16.1.1 <u>Annual Quality Systems Audit</u>

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given

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area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

# 16.1.2 <u>QA Technical Audits</u>

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

## 16.1.3 <u>SOP Method Compliance</u>

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

## 16.1.4 <u>Special Audits</u>

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

## 16.1.5 <u>Performance Testing</u>

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Non-potable Water, Soil and Hazardous Waste.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

# 16.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

# 16.2.1 Confidential Business Information (CBI) Considerations

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found within the 2003 NELAC standards.

## 16.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

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### SECTION 17

## MANAGEMENT REVIEWS (NELAC 5.4.14)

### 17.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Operations Manager, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

### 17.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director and QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals & objectives. Corporate Operations and Corporate QA personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the years that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management review (Corporate Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:

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- Adequacy of staff, equipment and facility resources.
- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

### 17.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

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#### **SECTION 18**

### PERSONNEL (NELAC 5.5.2)

### 18.1 <u>OVERVIEW</u>

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

# 18.2 EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL PERSONNEL

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

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Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, colony counting, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
GFAA, CVAA, FLAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – <u>General</u>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee
Technical Director/ Department Managers – <u>Wet Chem</u> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

## 18.3 <u>TRAINING</u>

The laboratory is committed to furthering the professional and technical development of employees at all levels.

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Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 20.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Laboratory Training SOP (TestAmerica Edison SOP No. ED-GEN-022).

## 18.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CA-L-P-001) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

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### SECTION 19

### ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

### 19.1 <u>OVERVIEW</u>

The laboratory is a 42,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

#### 19.2 <u>ENVIRONMENT</u>

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity and temperature levels in the laboratory (when appropriate).

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When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## 19.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

• Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

# 19.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

## 19.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

## SECTION 20

### TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

### 20.1 <u>OVERVIEW</u>

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

## 20.2 STANDARD OPERATING PROCEDURES (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

## 20.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

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The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

# 20.4 SELECTION OF METHODS

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

# 20.4.1 <u>Sources of Methods</u>

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- <u>Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act</u>, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water.<u>Revised as of July 1, 1995, Appendix</u> <u>A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)</u>
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- <u>Methods for the Determination of Inorganic Substances in Environmental Samples</u>, EPA-600/R-93/100, August 1993.
- <u>Methods for the Determination of Metals in Environmental Samples</u>, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- <u>Methods for the Determination of Organic Compounds in Drinking Water</u>, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. <u>Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series)</u> (EPA 500 Series methods)
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994

- <u>Statement of Work for Inorganics Analysis</u>, ILM04.1, USEPA Contract Laboratory Program Multimedia, Multi-concentration.
- <u>Statement of Work for Organics Analysis</u>, OLM04.2, USEPA Contract Laboratory Program, Multimedia, Multi-concentration.
- <u>Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLMO4.1, USEPA</u> Contract Laboratory Program, September 1998.
- <u>Standard Methods for the Examination of Water and Wastewater</u>, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup> edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- <u>Annual Book of ASTM Standards</u>, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

## 20.4.2 <u>Demonstration of Capability</u>

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

**20.4.2.1** A demonstration of capability (DOC) (reference TestAmerica Edison Training SOP No. ED-GEN-022) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

**20.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Department Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

**20.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 20.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

# 20.4.3 Initial Demonstration of Capability (IDOC) Procedures

**20.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**20.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**20.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**20.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**20.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**20.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**20.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 20.4.3.3 above.
- Beginning with 20.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 20-1 for an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

# 20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## 20.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

## 20.6.1 Method Validation and Verification Activities for All New Methods

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

### 20.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

### 20.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

### 20.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

#### 20.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

#### 20.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### 20.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

## 20.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

## 20.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

# 20.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. [To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate tvalue multiplier is used]

Refer to the Corporate SOP No. CA-Q-S-006 for details on the laboratory's MDL process.

## 20.8 INSTRUMENT DETECTION LIMITS (IDL)

**20.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**20.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

**20.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

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# 20.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

**20.9.1** Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

**20.9.2** When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

# 20.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

# 20.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

## 20.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

**20.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

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**20.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**20.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**20.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 +/- 0.5 mg/l.

**20.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 20.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also may be variables present (e.g., sample homogeneity, analyte precipitation over time, etc.) that affect the results of a reanalysis. Bearing these factors in mind, the laboratory will reanalyze samples at a client's request with the following caveats. (Note: Client specific Contractual Terms & Conditions for reanalysis protocols may supercede the following items).

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within ± 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

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• Due to the potential for increased variability, reanalysis may not be applicable to Nonhomogenous, Encore, and Sodium Bisulfate preserved samples. See the Laboratory Director if unsure.

## 20.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### 20.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in TestAmerica Edison SOPs No. ED-GEN-001 (Data Management and Handling Procedures) and ED-GEN-002 (Document Control). The laboratory is currently running the TALS LIMS which is a in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

- **20.14.1.1** <u>Maintain the Database Integrity:</u> Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.
- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.
- **20.14.1.2** Ensure Information Availability: Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.
- **20.14.1.3** <u>Maintain Confidentiality:</u> Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

#### 20.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

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Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- **20.14.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/<u>year</u>). It must be easily identifiable who performed which tasks if multiple people were involved.
- **20.14.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (μg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (μg/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.
- **20.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.
- **20.14.2.4** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- **20.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

#### 20.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample

ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 13.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Department Manager/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

### 20.14.4 <u>Review / Verification Procedures</u>

Review procedures are out lined in several SOPs (including but not limited to, TestAmerica Edison SOP Nos. ED-GEN-021: Data Review, ED-SPM-001:Login, and ED-RP-001:Reports Production) to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The general review concepts are discussed below, more specific information can be found in the SOPs.

- **20.14.4.1** The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.
- **20.14.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst or Department Manager/Supervisor performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Manual integrations are also electronically reviewed periodically by the QA Department utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:
  - QC data are outside the specified control limits for accuracy and precision
  - Reviewed sample data does not match with reported results
  - Unusual detection limit changes are observed
  - Samples having unusually high results
  - Samples exceeding a known regulatory limit
  - Raw data indicating some type of contamination or poor technique
  - Inconsistent peak integration

- Transcription errors
- Results outside of calibration range
- **20.14.4.3** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, Laboratory Operations Manager, or Department Manager for further investigation. Corrective action is initiated whenever necessary.
- **20.14.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.
- **20.14.4.5** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, chain of custody is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.
- **20.14.4.6** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.

## 20.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002).

- **20.14.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- **20.14.5.2** Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

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- **20.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- **20.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

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# Figure 20-1. Example - Demonstration of Capability Documentation

	DEMC	NSTRAT	ION OF	CAPABI	LITIY (DOC)	
Laboratory Name:						
Laboratory Address:						
Method:	A	4/->-	Matrix:			
Date: Source of Analyte(s):	Analys	st(s):	****			
Source of Analyte(s)						-
			Analytical F	Results		
Analyst Conc.	. (Units) Re	p1 Rep	2 Rep 3	Rep 4	Avg. % Recovery	% RSD
% RSD = Percent relativ	ve standard de	viation = sta	ndard devi	ation divide	ed by average % Recover	у
Raw data reference:						
Certification Statemen	ıt:					
We, the undersigned, co						
1. The cited test meth	od has met De	monstration	of Capabil	ity requirer	nents.	
2. The test method wa	as performed b	y the analys	t(s) identifi	ed on this ( Ps are ava	ailable for all personnel on	site
<ol> <li>A copy of the test n</li> <li>The data associat</li> </ol>	red with the n	hethod den	onstration	of capab	ility are true, accurate,	complete, and self-
explanatory.						
5. All raw data neces	sary to recons	truct and v	alidate the	se analyse	es have been retained at	the facility, and the
associated information i	s well organize	d and avail	able for rev	iew.		
6.						
Analyst Signature			Date			
Technical Director Sign	ature		Date			
Quality Assurance Coor	rdinator Signatu	ire	Date			

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### SECTION 21

### EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

### 21.1 OVERVIEW

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. An example laboratory equipment list is presented in Table 21-1. The most current list of laboratory instrumentation can be found in TestAmerica Edison Work Instruction No. ED-WI-002 (Equipment Inventory)

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

### 21.2 PREVENTIVE MAINTENANCE

**21.2.1** The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**21.2.2** Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

**21.2.3** Table 21-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

**21.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

**21.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement

of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

- **21.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- **21.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed may be affixed into the logbooks adjacent to pages describing the maintenance performed or filed in the Department Managers office If stapled into the logbook the stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

**21.2.5** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

**21.2.6** In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

**21.2.7** If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

# 21.3 <u>SUPPORT EQUIPMENT</u>

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

## 21.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

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Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

### 21.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to  $\pm$  0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

#### 21.3.3 <u>Thermometers</u>

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP No. ED-GEN-014 (Thermometer Calibration).

# 21.3.4 <u>Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators</u>

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between >  $0^{\circ}$ C and  $\leq 6^{\circ}$ C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

### 21.3.5 <u>Autopipettors, Dilutors, and Syringes</u>

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements. Refer to TestAmerica Edison SOP No. ED-GEN-011 (Calibration and Use of Lab Pippettes).

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

#### 21.3.6 <u>Autoclaves</u>

The laboratory utilizes autoclaves in the sample preparation step for certain mercury analysis procedures. These autoclaves have direct reading temperature and pressure gauges. These gauges are checked for accuracy on an annual basis.

### 21.3.7 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated as needed based on manufacturers recommendations.

## 21.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 13).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

## 21.4.1 CALIBRATION STANDARDS

**21.4.1.1** Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

**21.4.1.2** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

**21.4.1.3** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

**21.4.1.4** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

## 21.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification

applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

## 21.4.2.1 Verification of Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

# 21.4.2.2 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

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### 21.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

## 21.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Table 21-1. Example: Laboratory Instrumentation List						
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
METALS						
ICP	Thermo Jarrell Ash (1) S/N 341490	61E Trace	1994	Dec94	Yes	6010B, 200.7, CLP
	Thermo Jarrell Ash (2) S/N 356490	61E Trace	1998	Feb98	Yes	6010B, 200.7, CLP
	Thermo Jarrell Ash (3) S/N 493890	61E Trace	2000	Sep00	Yes	6010B, 200.7, CLP
	Thermo Jarrell Ash (4) S/N: ICP-20073407	ICAP 6500 Duo View	2007	TBD	Yew	6010B, 200.7, CLP
ICP-MS	Agilent Technologies S/N JP51201560 PolyScience	7500ce	2006	May06	Yes	6020A, 200.8
Heat Exchanger	S/N G57335 Cetac	3370				
Autosampler Mercury Analyzer	S/N 120536A520	ASX520	2002		Vac	74714 7470 046 1
Mercury Analyzer	Leeman Labs(3) S/N HA-3010	Hydra AA	2003	Jan04	Yes	7471A, 7470, 245.1 CLP
	Leeman Labs (4) S/N HA-4008	Hydra AA	2004	Jun04	Yes	7471A, 7470, 245.1 CLP
Hotblock 1	Environmental Express Limited S/N 2772CEC1378	SC154	2003	2003	No	3050B, CLP
Hotblock 2	Environmental Express Limited S/N 2391CEC1273	SC154	2004	2004	No	3050B, CLP
Autoclave (Out of Service)	Steril-Matic S/N 95-2678	MEA 109-85-E	1996	1996	No	7471A
Hot Plate 1 (Out of Service)	Fischer Scientific S/N 1000132		Jan04	Jan04	No	200.7, 3010A, 3020A, CLP
Hot Plate 2 (Out of Service)	Fischer Scientific S/N 1000153		Oct04	Oct04	No	200.7, 3010A, 3020A, CLP
Hot Plate 3 (Out of Service)	Fischer Scientific S/N 1000168		Jul03	Jul03	No	200.7, 3010A, 3020A, CLP
Hot Plate 4 (Out of Service)	Fischer Scientific S/N 1000169		May05	May05	No	200.7, 3010A, 3020A, CLP
Hot Plate 5 (Out of Service)	Fischer Scientific S/N 1000170		Apr05	Apr05	No	200.7, 3010A, 3020A, CLP
Hot Plate 6 (Out of Service)	Fischer Scientific S/N 1000203		Dec04	Dec04	No	200.7, 3010A, 3020A, CLP
Hot Plate 7 (Out of Service)	Fischer Scientific S/N 1000210		Apr05	Apr05	No	200.7, 3010A, 3020A, CLP
Hot Plate 8 (Out of Service)	Fischer Scientific S/N 1000220		Jun05	Jun05	No	200.7, 3010A, 3020A, CLP
Hotblock 3	Environmental Express Limited S/N 4298CEC2048	SC150	2004	2004	No	200.7, 3010A, 200.8, CLP
Hotblock 4	Environmental Express Limited S/N 4507CEC2115	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP

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Table 21-1.    Example: Laboratory Instrumentation List								
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed		
Hotblock 5	Environmental Express	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP		
	Limited S/N 4667CEC2183							
Hotblock 6	Environmental Express	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP		
	Limited							
Hotblock 7	S/N 4667CEC2183 Environmental Express	SC150	2006	2006	No	200.7, 3010A, 200.8, CLP		
HOLDIOCK /	Limited	30130	2000	2000				
	S/N 2772CDC1378		2005	0005	N1-	3050B, CLP		
Balance # 35	Acculab 18255989		2005	2005	No	3050B, CLP		
Balance # 33	Ohaus		2001	2001	No	7471A		
	F0461200521139					74710		
Autoclave	Steril-Matic S/N 201188	STME	2002	2002	No	7471A		
<u>GC/MS</u> <u>Semivolatiles</u>				4000	N/ I	00700 605		
<u>oomroidaleo</u>	Hewlett-Packard		1986	1986	Yes	8270C, 625		
	S/N 3223A43511	5971						
(BNAMS1/GC)	S/N 3118A02442	7673						
GC	S/N 3013A21967							
MS	S/N 3249A30680							
Tower	S/N 3249A30674							
Tray								
Controller	Hewlett-Packard		1986	1986	Yes	8270C, 625, CLP		
(BNAMS2/GC) GC	S/N 2618A07933		1900					
GC MS	S/N 2018A07933 S/N 3234A04110	5971						
Tower	S/N 2704A08901	7673A						
Tray	S/N 2718A08680							
Controller	S/N 2607A02892							
	Hewlett-Packard		1986	1986	Yes	8270C, 625, CLP		
(BNAMS3/GC) GC	S/N 3140A38366							
MS	S/N 3140A38386 S/N 3188A02926	5971						
Tower	S/N 3188A02926 S/N 3266A31274	7673						
Tray	S/N 3021A21499	1010						
Controller	S/N 3138A27180							
(BNAMS4/GC)	Hewlett-Packard		1986	1986	Yes	8270C, 625, CLP		
(BINAMIS4/GC) GC	S/N 3108A34490							
MS	S/N 3114A02077	5971A						
Tower	S/N 2546A02861	7673A						
Tray	S/N 2942A20598							
Controller	S/N 2803A11211							
(BNAMS5/GC)	Agilent Technologies		2007	2007	Yes	8270C, 625, CLP		
(BNAMS5/GC) GC	S/N CN10726100							
MS	S/N US35120328	5975C						
Tower	S/N CN72441261	7890A						
Tray	S/N CN40427800	1000/						
Controller	S/N CN40427800							

	Table 21	-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
(BNAMS6/GC)	Hewlett-Packard		1990	1990	Yes	8270C, 625, CLP
GC	S/N 3336A54722					
MS	S/N 3234A04274	5971				
Tower	S/N 2843A13155	7673				
Tray	S/N 2933A11253					
Controller	S/N 3018A21811					
BNAMS7/GC)	Hewlett-Packard		1990	1990	Yes	8270C, 625, CLP
GC	S/N 3235A45833					
MS	S/N 3307A00368	5972				
Tower	S/N 2546602130	7673A				
Tray	S/N 2633A02968					
Controller	S/N 2511A01985					
BNAMS8/GC)	Hewlett-Packard		1990	1990	Yes	8270C, 625, CLP
GC	S/N 336A56444		1000	1000		
MS	S/N 3435A01857	5972				
Tower	S/N C11144007149	A0C-20i				
Tray	S/N C11154103496	700-201				
Controller	S/N 626059SA					
			2004	2004	Yes	8270C, 625, CLP
BNAMS9/GC)	Agilent Technologies		2004	2004	res	02700, 020, 0LF
GC	S/N CN10349071	6070				
MS _	S/N US35120328	5973				
Tower	S/N CN35134357	7683				
Tray	S/N CN40427800					
Controller	S/N CN40427800					
BNAMS10/GC)	Agilent Technologies		2004	2004	Yes	8270C, 625, CLP
GC	S/N CN10403063					
MS	S/N US35120373	5973				
Tower	S/N CN40334758	7683				
Tray	S/N CN40327770					
Controller	S/N CN40327770					
BNAMS11/GC)	Agilent Technologies		2007	2007	Yes	8270C, 625, CLP
GC	S/N CN10727109					,,
MS	S/N US71236621	5975C				
Tower	S/N CN35134357	7890A				
Tray	S/N CN72441255	,				
Controller	5.1 011/211/200					
BNAGC2	Hewlett-Packard		1986	1986	Yes	Screen
	S/N 3336A55994	5890 II				2010011
GC	S/N 3336A55994 S/N 3004A20530	7673				
Tower 1 Tower 2		1013				
Tray	S/N 3613A21129					
Controller	S/N 3021A21938					
	S/N 3244A30371					

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	Table 21	-1. Example: L	aboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
BNAGC8	Hewlett-Packard		1986	1986	Yes	Screen
GC	S/N 3121A35833	5890				
Tower 1	S/N 2704805765	7673A				
Tray	S/N 3131A25914					
Controller	S/N 2921A03449					
Manifold Gases	Western Enterprise 28452	Innovator HBAC2-5-4	10/29/04	11/1/04	No	
GC/MS Volatiles					Yes	8260, 624, CLP, 524.2
VOANO	Agilent	5975	Feb06	Jul06		
VOAMS1	S/N US60532504	C000NI		Jul06		
GC	Agilent S/N CN10606023	6890N	Feb06	Juloo		
GC	OI	4551A	Feb06	Jul06		
Autosampler	S/N D60345B194	455TA	rebuo	50100		
Autosampier	OI	4660	Feb06	Jul06		
Concentrator	S/N D608466853	4000	1 6000	5000		
Concentrator	OI	SAM	Feb06	Jul06		
Spiker	S/N E610475713	0, 101	1 0.500			
VOAMS2	Hewlett-Packard	5975C	2008	2008	Yes	8260, 624, CLP,
VOANISZ	S/N US80838709	00100	2000	2000		,,
GC	Hewlett-Packard	7890A	2008	2008		
	S/N CN10813013					
Autosampler	EST	Archon 51	2008	2008		
· · · · · · · · · · · · · · · · · · ·	S/N 15264					
Concentrator	EST	Encon Evolution	2008	2008		
	S/N 104041408					
VOAMS3	Agilent	5973inert	Feb04	Aug04	Yes	8260B, 624, CLP, 524.2
	S/N US35120382					
GC	Agilent	6890N	Feb04	Aug04		
	S/N CN10406105					
Autosampler	EST	Centurion	Jun04	Aug04		
	S/N CENT140051304					
Concentrator A	EST	Encon	May04	Aug04		
	S/N 367060704					
Concentrator B	EST	Encon	May04	Aug04		
	S/N 368060704					

	Table 21	-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
VOAMS4	Hewlett-Packard S/N US80838712	5975C	2008	2008	Yes	8260, 624, CLP,
GC	Hewlett-Packard	7890A	2008	2008		
Autosampler 1	S/N CN10813014 OI	4552	2008	2008		
	S/N 15266 Ol					
Concentrator	S/N D809466076	2008	2008	2008		
VOAMS5	Hewlett-Packard	5971	1996	1996	Yes	8260B, 624, CLP, 524.2
	S/N 3234A04198					
GC	Hewlett-Packard S/N 3033A33368	5890 II	1996	1996		
Autosampler	<b>Archon</b> S/N 11957-696A	5100A	1996	1996		
Concentrator	OI S/N D310219	4560	1996	1996		
VOAMS6	Agilent VOAMS6 S/N US35120322	5973inert	Feb04	Apr04	Yes	624, 524.2, CLP
GC	Agilent S/N CN10406076	6890N	Feb04	Apr04		
Autosampler	OI S/N D54645B461	4551A	Nov05	Dec05		
Concentrator	OI S/N D548466579	4660	Nov05	Dec05		
Spiker	OI S/N C425475656	SAM	Jun04	Jul04		
VOAMS7	Agilent S/N US43110514	5973inert	Oct 04	Nov 04	Yes	624, 524.2,8260 CLP
GC	Agilent S/N CN10437064	6890N	Oct 04	May 06		
Autosampler	Teledyne Tekmar S/N US08121007	Solatek	Tekmar swap	May 08		
Concentrator	Teledyne Tekmar S/N US08007007	Stratum	Tekmar swap	May 08		
VOAMS8	Hewlett-Packard S/N 3118A02630	5971	1998	1998	Yes	8260B, 624, CLP, 524.2
GC	Hewlett-Packard S/N 3126A36935	5890 II	1998	1998		
Autosampler	EST Archon	5100A	1998	1998		
Concentrator	S/N 12206 OI S/N I418460464	4560	1998	1998		

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	Table 21	-1. Example:	Laboratory li	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Perform
VOAMS9	Hewlett-Packard	5971	1998	1998	Yes	8260B, 624, CLP, 5
	S/N 3118A03332					
GC	Hewlett-Packard	5890 II	1998	1998		
	S/N 3203A40292					
Autosampler	EST Archon	5100A	1998	1998		
	S/N 12207					
Concentrator	01	4560	1998	1998		
	S/N C302089					
VOAMS10	Hewlett-Packard	5972	1997	July /2000	Yes	8260, 624, CLP, 53
	S/N 3307A00392		(Whippany acquisition)	(In Edison)		
GC	Hewlett-Packard	5890	Unknown	1997		
	S/N 2728414257			(In Whippany)		
Autosampler	Teledyne Tekmar	Aquatek 70	Mar06			
	S/N 94312017			May 2008		
Concentrator	Tekmar	3000	1997			
	S/N 94087010					
VOAMS11	Agilent	5973N	Jun03	Jul03	Yes	8260B, 624, CLP, 5
	S/N US30965664					
GC	Agilent	6890N	Jun03	Jul03		
	S/N CN10324011					
Autosampler	EST Archon	5100A	Jun03	Jul03		
Concentrator	S/N 13970					
Concentrator	EST	Encon	Jun03	Jul03		
	S/N 279061703					

	Table 21-'	1. Example: L	aboratory Ir	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
	Agilent	5973inert	Oct04	Nov04	Yes	8260, 624, CLP, 524.2
VOAMS12	S/N US43110519					
	Agilent	6890N	Oct04	Jun05		
GC	S/N CN10439051					
	EST	Archon 5100A	May05	Jun05		
Autosampler	S/N 14448					
	EST	Encon	May05	Jun05		
Concentrator	S/N 430051605					
	Agilent	Performance	Jun05	Jun05		
Turbo Pump Upgrade	S/N 56115832					
	Agilent	5973inert	Oct04	Nov04	Yes	8260, 624, CLP, 524.2
VOAMS13	S/N US43110517					
	Agilent	6890N	Oct04	Jun05		
GC	S/N CN10439052					
	EST	Archon 5100A	May05	Jun05		
Autosampler	S/N 14449					
	EST	Encon	May05	Jun05		
Concentrator	S/N 431051605					
	Agilent	Performance	Jun05	Jun05		
Turbo Pump Upgrade	S/N 56069171					
Balance #22	Mettler 2115517886	PB1501	1997	1997	No	8260, 8015 GRO
	Ohaus	Explorer Pro	2006	2006	No	8260, 8015 GRO
Balance #50	1125573353					
Balance # 103	Denver Instruments 126008		2009	2009	No	8260
Oven Drying	Fisher Isotemp Oven 502N0045	13-246-516G	2/15/2005	3/3/2005	NO	
Oven Drying	Baxter 199012	DX-1	2000	2000	No	

Table 21-1.    Example: Laboratory Instrumentation List							
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed	
GC Volatiles					Yes	8015B (GRO)	
	Agilent	6890N	Mar06	May06			
GC1	S/N US10610006 OI	4552	Feb06	May06			
Autosampler	S/N 14608	4002	1 0000	Widyoo			
	OI	4660	Feb06	May06			
Concentrator	S/N D607466340 OI	4551A	Feb06	May06			
Autosampler	S/N D60745B342	43317	1 6000	Mayoo			
	OI	4660	Feb06	May06			
Concentrator	S/N D607466341 OI	SAM	Feb06	May06			
Spiker	OI S/N E610475713	SAM	rebud	iviay00			
			1000	4000	Yes	Carooning/2010	
GC2	Hewlett-Packard S/N 2921A23492	589011	1993	1993	Yes	Screening/3810	
Autosampler 1	Tekmar	7050	Jun04	Jul04			
	S/N US04156005						
Headspace 1	Tekmar S/N US04156003	7000	Jun04	Jul04			
Autosampler 2	Tekmar	7050	Jun04	Jul04			
	S/N US04148014						
Headspace 2	Tekmar S/N US04163001	7000	Jun04	Jul04			
GC3	Hewlett-Packard	589011	1996	1996	Yes	8015B (GRO)	
	S/N 3310A49242						
PID	OI S/N 01 1107	4430	1996	1996			
Autosampler	S/N 91-I107 Dynatech Archon	5100	1996	1996			
	S/N 11780-795						
Concnetrator	OI	4560	1996	1996			
SCREEN1/2 GC	S/N J437460274 Hewlett-Packard	5890 II	1989	1989	Yes	Screening	
CORLENT/2 CO	S/N 2950A29246						
Autosampler 1	Tekmar	7050	1989	1989			
Headspace 1	S/N 91025014 Tekmar	7000	1989	1989			
neauspace i	S/N 91163066	1000	1000				
Autosampler 2	Tekmar	7050	1989	1989			
Llanderson O	S/N 91168012	7000	1989	1989			
Headspace 2	Tekmar S/N 90255003	7000	1909	1909			

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	Table 21	-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
SCREEN3/4 GC	Hewlett-Packard	5890	1998	1998	Yes	Screening/3810
	S/N 2908A21857					
Autosampler 1	Tekmar	7050	1998	1998		
	S/N 91346013					
Headspace 1	Tekmar	7000	1998	1998		
	S/N 91339015					
Autosampler 2	Tekmar	7050	1998	1998		
	S/N 90256011		1000			
Headspace 2	Tekmar	7000	1998	1998		
	S/N 91025010		4000			
H-Nu PID	H-nu Systems	PI101	1989	1989	No	Headspace Screening
	S/N 801023					
Hood					No	
Ductless Fume	Air Science					
	P41007	PurAir15	Oct04	Nov04		
GC Semivolatiles	Agilent Technologies		2003	2005	Yes	NJDEP-OQA-QAM-025
BNAGC1	S/N US10248079	6890N				
GC Network	S/N CN24428026	G2613A				
Injector Module	S/N CN24322270	G2614A				
Tray			1987			
BNAGC3 GC Network	Hewlett Packard		1987	1987	Yes	GC Fingerprints
Tower	S/N 2643A12162	5890 II				
Tray	S/N C11144007157KG					
	S/N C11154003268KG		- Fab06			
BNAGC4 GC Network	Agilent Technologies		Feb06	Apr06	Yes	8015B DRO/Fingerprints
Injector Module 1	S/N US10610005	6890N				QAM-025
Injector Module 1	S/N CN43820808	G2913A				
-	S/N CN43820804	G2914A				
Tray	S/N CN43830663	G2614A	1997			
BNAGC5	Hewlett-Packard		1997	1997	Yes	8015B Alcohols
GC	S/N 2728A14513	5890				
Tower		7673				
Tray	S/N 2920A10887					
Controller	S/N 01866		1997	4007	N	004ED A
BNAGC6	Hewlett-Packard	5000 1	1997	1997	Yes	8015B Amines
GC	S/N 3203A40054	5890 II				
Tower 1	S/N 3120A28315	7673				
Tower 2	S/N 3202A27987					
Tray	S/N 3228A29094					
Controller	S/N 3138A27180					

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	Table 21-1. Example: Laboratory Instrumentation List							
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed		
BNAGC7	Hewlett-Packard		1999	1999	Yes	8015B Glycols		
GC	S/N 2443A03923	5890						
Tower 1	S/N 2546A02013	7673A						
Tray	S/N 2718A05293							
Controller	S/N 2929A15891							

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	Table 21	-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Pest/PCB						
	Hewlett-Packard		1992	1992	Yes	8081, CLP
GC1	S/N 2612A07669	5890A				
GC Mainframe	S/N CN22321930	G1513A				
Injector Module	S/N CN00005085	G1512A				
Controller	S/N US72101578	18596C				
Tray						
GC2	Hewlett-Packard		1992	1992	Yes	OUT OF SERVICE
GC Mainframe	S/N 2750A15933	5890A				
Injector Module 1	S/N 2932A14269	18593A				
Injector Module 2	S/N 2704A8875	1859 <b>3A</b>				
Controller	S/N 2749A09358	18594A				
Tray	S/N 2718A08934	18596A				
GC3	Hewlett-Packard		1992	1992	Yes	Herbicides
Series II GC	S/N 3223A42873	5890A				
Injector Module	S/N 3228A31372	1859 <b>3B</b>				
Controller	S/N 3049A23890	18594B				
Tray	S/N 3202A27453	18596 <b>B</b>				
GC4	Hewlett-Packard		1997	1997	Yes	8081
Series II Plus GC	S/N 336A54563	5890A				
Injector Module	S/N 3013A22344	18593B				
Controller	S/N 3227A29129	18594B				
Tray	S/N 3624A42191	18596 <b>B</b>				
GC5	Agilent Technologies		2002	2002	Yes	8081
GC Network	S/N US10226033	6890N				
Injector Module	S/N CN22025340	G2613A				
Tray	S/N CN21420543	G2614A			<u> </u>	
GC6	Hewlett-Packard		1998	1998	Yes	608
GC Mainframe	S/N 2950A26642	5890A				
Injector Module	S/N CN13420438	G1513A				
Controller	S/N CN00004777	G1512A				
Tray	S/N US20407961	18596 <b>C</b>			ļ	
GC7	Hewlett-Packard		1998	1998	Yes	8082
GC Mainframe	S/N 3029A29927	5890A				
Injector Module	S/N C11144007141	18593A				
Controller	S/N 626059	18594A				
Tray	S/N C11154103504	18596A				
GC8	Agilent Technologies		2000	2000	Yes	8082
GC Plus	S/N US00004463	6890				
Injector Module	S/N CN15221154	G151 <b>3A</b>				
Controller	S/N 3631A05939	G1512A				
Tray	S/N 3050A23572	18596C				

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	Table 2'	I-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
GC9	Agilent Technologies		2001	2001	Yes	8082
GC Plus	S/N US00043694	6890				
Injector Module	S/N CN13420437	G1513A				
Controller	S/N CN00004150	G1512A				
Tray	S/N US13807350	18596 <b>C</b>				
GC11	Agilent Technologies		2003	2003	Yes	CLP
GC Plus	S/N US00008746	6890				
Injector Module	S/N US64600228	G2513A				
Controller	S/N US72202100	G2512A				
Tray	S/N US22408138	18596 <b>C</b>				
WET CHEMISTRY						
Spectrophotometer	HACH S/N 1205122	DR2800	2007	2007	No	365.2, 7196A, 353.2, 410.4
Spectrophotometer	HACH S/N 1204684	DR2800	2007	2007	No	365.2, 7196A, 353.2, 410.4
Spectrophotometer	HACH S/N 11204422	DR2800	2007	2007	No	7196A, USGS
Turbidimeter	HF Scientific S/N 200604033	Micro 100	2006	2006	No	180.1, SM 2130B
Ion Selective Meter	Orion S/N 006825	720A	1994	1994	No	350.1+ .2, 340.2, 150.1
Ion Selective Meter	Orion S/N 092904	720A+	2007	2007	No	350.1+ .2, 340.2, 150.1
pH Meter	Orion S/N 010005	320	2002	2002	No	Cr6+
pH Meter	Orion S/N 009986	320	2002	2002	No	350.1/4500 NH3 H
pH Meter	Orion 320 S/N 016995	320	2002	2002	No	TCLP (1311)
pH meter	Orion 320 S/N 017414 VWR	320	0001	2009	No	4500-H B
Oven	S/N 0402001	1520	2001	2001		2540C
Oven	VWR	1300U	2001	2001	No	2540C
Oven	VWR	1305U	2001	2001	No	2540B
Oven	Fisher	230G	1997	1997	No	2540B, 2540D
Oven (Muffle Furnace)	Fisher S/N 901N002	550-14	2002	2002	No	160.4
Oven drying	VWR	1320	2001	2001	No	
Balance #27	A&D 12315883	HR-200	2005	2005	No	Gen. chem.
Balance #29	A&D 12315872	HR-200	2005	2005	No	160.1, 160.2
Balance #26	Sartorius 3503054	1712MP8	2003	2003	No	Gen. chem.
Balance #51	Ohaus 7125010794	Scout Pro	2006	2006	No	1311 (TCLP), 3060A
Balance #100	Mettler 122423439		2006	2006	No	Lloyd Kahn (TOC)
Balance # 101	Denver Instrument 126009		2009	2009	No	Gen. chem.

Instrument Type	Manufacturer	Model	Purchase	Install Date	Autosampler	Method Performed
instrument Type	manuracturer	moder	Date	instan Date	Autosampici	
Water Bath	Precision S/N 9302-112	50	1995	1995	No	7196A
Water Bath	Precision S/N 9305-024	50	1995	1995	No	7196A
Water System (Log-in)	Millipore S/N 07348-C		1990	1990	No	
Water System (Extr. room)	Barnstead S/N 1191020210415	D119 <b>11</b>	1995	1995	No	
FTIR	Perkin Elmer S/N 139038	1600	1991	1991	No	418.1
Printer	Epson S/N 61P107612	FX-870	2003	2003	No	418.1
Fixed IR	Buck Scientific S/N 1026	404	2004	2004	No	418.1
COD reactor	HACH S/N 980300017418	45600	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 900402268	45600	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 1202323	DRB 200	2007	2007	No	410.4, 5220D
COD reactor	HACH S/N 1209887	DRB 200	2007	2007	No	410.4, 5220D
Auto-analyzer	Lachat S/N A83000	QUICKEM 8000	1997	1997	Yes	335.3, 420.2, 353.2, 351.2, 350.1+ .2
Auto-analyzer	Lachat S/N 8300-1658	8000 Series	2000	2000	Yes	335.3, 350.1+ .2
TOC Analyzer	Shimadzu S/N 31242909	TOC <b>5000</b>	1997	1997	Yes	Lloyd Kahn's method, 415.1, 9060, 5310B
Autosampler	Shimadzu S/N 31816800	ASI-5000	1997	1997	Yes	415.1, 5310B, 9060
Solid Sample Module (1)	Shimadzu S/N 31303115	SSM-5000A	1997	1997	No	Lloyd Kahn's method
TOC Soil Analyzer (2)	Thermo Electron Corp. S/N 20034945	Flash EA 1112 Series	2004	2004	Yes	Lloyd Kahn's method
Printer	Epson S/N 41NE28676	LQ570	1997	1997	No	415.1
TOC Analyzer	Shimadzu S/N H51104335164	TOC-VCSH	2006	2006	Yes	Lloyd Kahn's method, 415.1, 9060, 5310B
Autosampler	Shimadzu S/N H52104301656SA	ASI-V	2006	2006	Yes	415.1, 5310B, 9060
Solid Sample Module	Shimadzu S/N H52504300040NK	SSM-500A	2006	2006	Yes	Lloyd Kahn's method
BOD Meter	YSI S/N 97S0534AE	5000	1998	1998	No	405.1
Incubator	GCA Precision Scientific		1998	1998	No	405.1
Hot Plate	Fischer Scientific S/N 103N0071		2001	2001	No	365.2
Hot Plate	Corning S/N 370301092774	PC-400	2007	2007	No	1311
Hot Plate	Fischer Scientific S/N 390502148495	PC-420	2007	2007	No	Lloyd Khan Method
Hot Plate	Fischer Scientific S/N 220897070707	PC-620	2007	2007	No	351.2
Conductivity Meter	Fischer Scientific S/N AB 81209007	Accumetab30	2002	2002	No	120.1, 9050A

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	Table <b>21</b> -	-1. Example: La	boratory l	nstrumentati	on List	·
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Vortex mixer	Thermolyne S/N 632000855604	M63215	2002	2002	No	351.2
Dishwasher	Miele Professional S/N 208479	G7783CD	2003	2003	No	Glassware
Easy-Dist Distillation	Westco S/N 1095		2003	2003	No	350.1+ .2, 420.2, 9066
Easy-Dist Distillation	Westco S/N J097		2003	2003	No	335.3, 9012A & B
Easy-Dist Distillation	Westco S/N 1063		2007	2007	No	350.1+.2, 420.2, 353.3, 9012A&B
Easy-Dist Distillation	Westco S/N 1110		2007	2007	No	353.3, 420.2, 9066
Discreet Analyzer (1)	Konelab S/N S2019177	20	2003	2003	Yes	Automated Wet Chem
Discreet Analyzer (2)	Konelab S/N 2519236	20	2003	2003	Yes	Automated Wet Chem
Dell Computer	Dell S/N 246175		2003	2003	No	Automated Wet Chem (Konelab)
BOD Aerator	Thomas Scientific S/N 1187	DOA-P104d-AA	1998	1998	No	405.1
BOD Plus Assay Liquid Handler DO meter YSI 52	Mantech Assoc., Inc. S/N 27OC3XB215 S/N 03C0812 AM	221 & 222 52CE	2003	2003	Yes	405.1
PC-Titration Plus Autotitrator Interface Titra-Rinse 1 Titra-Rinse 2 Buret Module 1 Buret Module 2 Titration Module	Mantech Assoc., Inc S/N MS-0H4-373 S/N MS-0G4-198 S/N MS-0G4-200 S/N MS-0H4-627 S/N MS-0H4-625 S/N MS-0B5-657	PC-1000-102/4 PC-1000-408 PC-1000-408 PC-1104-00 PC-1104-00 PC-1300-475	2004	2004		310.1, 2320B – Alkalinity 2320B – Carbonate, Bicarbonate 4500 CO2D – Carbon Dioxide 130.2, 2340C – Hardness
Ion Chromatograph Pump #1 Pump #2 Conduct. Detector Injector & Oven 2-Ch Interface Liq. Handling #1 Liq. Handling #2 Dil. Autosampler	Metrohm Peak, Inc. S/N 04187 S/N 04197 S/N 03181 S/N 04147 S/N 04144 S/N 04154 S/N 04158 S/N 04118 S/N 03198	818 818 819 820 830 833 833 833 838	May05	May05	Yes	7199
Filter pump	Emerson S/N SA55-NXGTB 4142		1997		No	Sample Filtering
Filter pump	Emerson S/N G8ECX	SA55JXgtd-4144	2002	2002	No	Sample Filtering
Redox meter	VWR S/N 001149	8005	1997	1997	No	SM2580
Rotator 1	AP & R Machine & Tool		2003	2003	No	600/8000/CLP
Rotator 2	S/N 222307 AP & R Machine & Tool S/N 222306		2003	2003	No	600/8000/CLP
Rotator 3	AP & R Machine & Tool S/N 222305		2003	2003	No	600/8000/CLP

	Table 21-1	. Example: L	aboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Rotator 4	AP & R Machine & Tool S/N 222304		2003	2003	No	600/8000/CLP
Rotator 5	AP & R Machine & Tool S/N 222303		2003	2003	No	600/8000/CLP
Rotator 6	AP & R Machine & Tool S/N 222302		2003	2003	No	600/8000/CLP
TCLP Extraction1 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1352	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction2 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1053	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction3 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 1249	3740-12 BRE	1997	1997	No	1311 TCLP, ZHE
TCLP Extraction4 Apparatus/Timer included	Environmental Express Limited S/N 3384-12-473	LE 1002	May05	May05	No	1311 TCLP, ZHE
TCLP Extraction5 Apparatus/Timer included	Environmental Express Limited S/N 3384-12-472	LE 1002	May05	May05	No	1311 TCLP, ZHE
TCLP Extraction6 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 2125	3740-12 BREII	Jul06	Sep06	No	1311 TCLP, ZHE
TCLP Extraction7 Apparatus/Timer included	Assoc. Design and Mfg. Co. S/N 2126	3740-12 BREII	Jul06	Sep06	No	1311 TCLP, ZHE
SAMPLE LOGIN Balance #13	Satorius	LC421	1995	1995	No	%Solids
Balance #104	S/N 50709085 Denver Instruments		2009		No	
Isotemp Oven 1	S/N 126006 Fisher S/N 410B01117	637G	Mar05	Mar05	No	%Solids
Isotemp Oven 2	Fisher S/N 505N0063	637G	Jun05	Jun05	No	%Solids
ORGANIC EXTRACTIONS						
N-EVAP #1	Organomation S/N 51004	8125	2004	2004	No	600/8000/CLP
N-EVAP #2	Organomation S/N 10253	N-EVAP 112	1990	1990	No	600/8000/CLP
Water Bath #1	Fisher Scientific S/N 605021280	15-491	2005	2005	No	600/8000/CLP
Water Bath #2	Fisher Scientific S/N (204272)	15-491	2007	2007	No	600/8000/CLP
Sonicator #1	Sonic & Material, Inc. S/N 3353027	VCX 500	2006	2006	No	8000/CLP

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Instrument Type	Manufacturer	Model	Purchase	Install Date	Autosampler	Method Performed
instrument Type	Manufacturer	model	Date			
Sonicator #2	Sonic & Material, Inc. S/N 3353028	VCX 500	2006	2006	No	8000/CLP
Sonicator #3	Tekmar S/N 7918	TM500	1990	1990	No	8000/CLP
Sonicator #4 share controller vith son #3)	Tekmar S/N 7918	TM500	1990	1990	No	8000/CLP
Sonicator #5	Sonic & Material, Inc. S/N 41748	VCX 500	2004	2004	No	8000/CLP
Sonicator #6	Sonic & Material, Inc. S/N 41755	VCX 500	2004	2004	No	8000/CLP
Muffle Furnace #1	Thermolyne S/N 40800875	F6010	1990	1990	No	600/8000/CLP
Muffle Furnace #2	Thermolyne S/N (warn out)	F6028C	1990	1990	No	600/8000/CLP
Large Muffle Furnace	Wilt Industries S/N 041213	210	2001	2001	No	600/8000/CLP
Dishwasher #1	Miele Professional S/N 53075564	G7783CD	2003	2003	No	608/8000/CLP
Dishwasher #2	Miele Professional S/N 53075571	G7783CD	2003	2003	No	608/8000/CLP
Vacuum Pump #1	Emerson electric MLD S/N UNL231171	5KH36KN90HX	1990	1990	No	600/8000/CLP
Vortex	Scientific Industries S/N 2-318564	6560	1995	1995	No	600/8000/CLP
Electric Mixer	Barnstead/Thermolyne S/N 125404091646		1995	1995	No	600/8000/CLP
Mini Hotplate/Stir	VWR Scientific S/N 33918-604	220	1995	1995	No	600/8000/CLP
Centrifuge #1	Sigma S/N 78646	2-5	2001	2001	No	600/8000/CLP
Centrifuge #2	Sigma S/N 78647	2-5	2001	2001	No	600/8000/CLP
Centrifuge #3 (Out of Service)	Sigma S/N 80226	2-5	2001	2001	No	600/8000/CLP
Balance # 60	Ohaus S/N 7125471186	Scout Pro	2007	2007	No	600/8000/CLP
Balance #28	A&D S/N 12315879	HR-200	2005	2005	No	600/8000/CLP
Balance #30	A&D S/N 12315880	HR-200	2005	2005	No	600/8000/CLP
Soxtherm 1	Ol Analytical S/N 4012358	Type 07-5101	2002	2002	No	8000
Controller Chiller						

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	Table 21-	1. Example: L	aboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Soxtherm 2	Ol Analytical S/N 4010018	Type 07-5101	2002	2002	No	8000
Controller Chiller	S/N 4010088 S/N 10200022					
Soxtherm 3	Ol Analytical S/N 4012359	Туре 07-5101	2002	2002	No	8000
Controller Chiller	S/N 4002805 S/N 10365037					
Soxtherm 4	Ol Analytical S/N 429023	Type 07-5101	2002	2002	No	8000
Controller Chiller	S/N 4022012 S/N 101365037					
Soxtherm 5	Gerhardt S/N 4073032	SOX 416	2007	2007	No	8000
Controller Chiller	S/N 4051753 S/N 107344070 (Thermo)					
Soxtherm 6	Gerhardt S/N 4073033	SOX 416	2007	2007	No	8000
Controller Chiller	S/N 4051753 S/N 107344070 (Thermo)					
Soxtherm 7	Gerhardt S/N 4073030	SOX 416	2007	2007	No	8000
Controller Chiller	S/N 4051753 S/N 107344069 (Thermo)					
Soxtherm 8	Gerhardt S/N 4073031	SOX 416	2007	2007	No	8000
Controller Chiller	S/N 4051753 S/N 107344069 (Thermo)					
Soxtherm 9	OI Analytical S/N 4012357	Type 07-5101	2003	2003	No	8000
Controller Chiller	S/N 4012357 S/N 4012354 S/N 101361126					
Soxtherm10	OI Analytical S/N 4010016	Туре 07-5101	2003	2003	No	8000
Controller Chiller	S/N 4010010 S/N 4012353 S/N 101361126					

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Inscriment Type         Inscriment of the second secon		Table 21	-1. Example: L				
SN 4012356 SN 480017 SN 4002024         Type 07-5101 SN 403002         2005 Southerm 12         No         8000 S000           Controller Chiller         SN 4003530 SN 4033530 SN 4031547         Type 07-5101 SN 40302024         2005         2006         No         8000           Southerm 13 Chiller         Genardt SN 4031667 SN 4031667 SN 4031667         SOX416         2006         2006         No         8000           Southerm 14         Genardt SN 4031667         SOX416         2006         2006         No         8000           Southerm 14         Genardt SN 4031667         SOX416         2006         2006         No         8000           Southerm 14         Genardt SN 4051747 SN 10381124         SOX416         2006         2006         No         8000           Southerm 15         Genardt SN 405138124         SOX416         2006         2006         No         8000           Southerm 15         Genardt SN 4051342         SOX416         2008         2006         No         8000           Southerm 16         Genardt SN 4051747         SOX416         2006         2006         No         8151           Mrist Action Shaker 2         Burrell         75         2003         2003         No         8151           Syn 405174	Instrument Type	Manufacturer	Model		Install Date	Autosampler	Method Performed
Onlifer         SN 102002024         Image: Controller SN 403350         Type 07-5101         2005         No         8000           Soxtherm12         Controller SN 403350         SN 401812         SN 10200024         SN 403350         SN 403350         SN 403350         SN 403507         SN 40507         SN 405077         SN 40507         SN 405077         SN 40507         SN 405077         SN 4	Soxtherm11		Type 07-5101	2005	2005	No	8000
Outschert         Operating and the second seco							
Controller Chiller         S/N 401812 S/N 102002024         SOX416         2006         2006         No         8000           Soxtherm13         Gerhardt S/N 4031667         SOX416         2006         2006         No         8000           Controller Chiller         S/N 4031747         S/N 10361121         1177PD         SOXtherm 14         Gerhardt         SOX416         2006         2006         No         8000           Soxtherm 14         Gerhardt         SOX416         2006         2006         No         8000           Soxtherm 15         Gerhardt         SOX416         2006         2006         No         8000           Soxtherm 15         Gerhardt         SOX416         2006         2006         No         8000           Soxtherm 16         Gerhardt         SOX416         2006         2003         No         8151           1         S/N	Soxtherm12		Type 07-5101	2005	2005	No	8000
Controller Controller         SiN 4031667 SiN 4031667 SiN 4031667 SiN 4031666         SOX416         2006         2006         No         8000           Soxtherm 14         Gerhardt SiN 4031666         SOX416         2006         2006         No         8000           Soxtherm 15         Gerhardt SiN 4051747         SOX416         2006         2006         No         8000           Soxtherm 15         Gerhardt SiN 4051747         SOX416         2006         2006         No         8000           Soxtherm 15         Gerhardt SiN 4051747         SOX416         2006         2006         No         8000           Soxtherm 16         Gerhardt SiN 4051747         SOX416         2006         2006         No         8000           Soxtherm 16         Gerhardt SiN 4051747         SOX416         2006         2006         No         8000           Wrist Action Shaker 2         Burrell SiN 12910443         75         2003         2003         No         8151           Titt Action Shaker 2         Labline SiN 12910443         3589         2002         2000         No         8151           FIELD SERVICES pH/Temp meter         Thormo Orion 15035         250A +         2000         2000         No         Dissolved Oxyge							
SN 1013611211177PDImage: sector of the	Soxtherm13		SOX416	2006	2006	No	8000
Control In 11         Control of C			1177PD				
Soxtherm 15Gerhardt S/N 4051583 S/N 40501747 S/N 4050017 (VWR)SOX41620062006No8000Soxtherm 16Gerhardt S/N 4051747 S/N 4051747 S/N 4051747 S/N 10650017 (VWR)SOX41620062006No8000Wrist Action Shaker 2Burrell S/N 10650017 (VWR)S58920032003No8151Wrist Action Shaker 2Labline S/N 10650017 (VWR)358920032003No8151Wrist Action Shaker 2Labline S/N 12910443358920032003No8151FIELD SERVICES pH/Temp meterThermo Orion 15035250A+20002002NoConductivity Do meterDO meterHACH 01200001321Sension 620022002NoDissolved OxygeDo meterHACH 01200003552Sension 620002000NoDissolved OxygeTurbidity meterLa Motte 0119099720202002NoTurbidityTurbidity meterLa Motte 0119099720202002NoTurbidity	Soxtherm 14	S/N 4031666 S/N 4051747	SOX416	2006	2006	No	8000
Soxtherm 16 Si/N 4051582 Si/N 4051747 Si/N 10650017 (VWR)Gerhardt Si/N 4051747 Si/N 10650017 (VWR)SOX41620062006No8000Wrist Action Shaker 1Burrell Si/N7520032003No8151Mrist Action Shaker 2Labline Si/N 12910443358920032003No8151FIELD SERVICES pH/Temp meterThermo Orion 15035358920032000No8151Conductivity meterHACH 21000005660250A+20002000NoPH, TemperatureDO meterHACH 0200001321Sension 620022002NoDissolved OxygeDo meterHACH 01200002352Sension 620002000NoDissolved OxygeTurbidity meterLa Mote 01490002352Sension 620002000NoDissolved OxygeTurbidity meterLa Mote 01490002352202019981998NoTurbidityTurbidity meterLa Mote 0149000235220202000NoTurbidity	Soxtherm 15	Gerhardt S/N 4051583 S/N 4051747	SOX416	2006	2006	No	8000
Wrist Action Shaker 1Burrell S/N7520032003No8151Wrist Action Shaker 2Labline S/N 12910443358920032003No8151FIELD SERVICES pH/Temp meterThermo Orion 15035250A+20002000NoPH, TemperatureConductivity meterHACH 21000005660Sension 520022002NoConductivityDO meterHACH 001200002352Sension 620022002NoDissolved OxygeTurbidity meterLa Motte 0119-0997202019981998NoTurbidityTurbidity meterLa Motte 3897-510220202002NoTurbidity	Soxtherm 16	Gerhardt S/N 4051582 S/N 4051747	SOX416	2006	2006	No	8000
Wrist Action Shaker 2Labline S/N 12910443358920032003No8151FIELD SERVICES pH/Temp meterThermo Orion 15035250A+20002000NoPH, TemperatureConductivity meterHACH 2100005660Sension 520022002NoConductivityDO meterHACH 0200001321Sension 620022002NoDissolved OxygeDO meterHACH 		Burrell	75	2003	2003	No	8151
pH/Temp meterThermo Orion 15035250A+20002000NopH, TemperatureConductivity meterHACH 21000005660Sension 520022002NoConductivityDO meterHACH 0200001321Sension 620022002NoDissolved OxygeDO meterHACH 001200002352Sension 620002000NoDissolved OxygeTurbidity meterLa Motte 0119-0997202019981998NoTurbidityTurbidity meterLa Motte 3897-5102202020022002NoTurbidity		Labline	3589	2003	2003	No	8151
Conductivity incluitDistributionConstraintCons			250A+	2000	2000	No	pH, Temperature
DO meterHACH 0200001321Sension 620022002NoDissolved OxygeDO meterHACH 001200002352Sension 620002000NoDissolved OxygeTurbidity meterLa Motte 0119-0997202019981998NoTurbidityTurbidity meterLa Motte 3897-5102202020022002NoTurbidity	Conductivity meter		Sension 5	2002			-
Do fileder         Do fileder <thdo fileder<="" th="">         Do fileder         Do filed</thdo>	DO meter	НАСН					Dissolved Oxygen
Turbidity meterLa Motte 0119-0997202020022002NoTurbidityTurbidity meterLa Motte 3897-5102202020022002NoTurbidity		001200002352					
3897-5102	-	0119-0997					
$\pm 120170$ $1 2007 1 2007 1 000 10000000000000000000$	_	3897-5102	2020	2002	2002	No	Turbidity
3649-3802	-	3649-3802		2002			pH, Oxidation reduction
643409		643409		2005	2005		pH, Oxidation reduction

	Table 21	-1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	<b>Ma</b> nufa <b>ctur</b> er	Model	Purchase Date	Install Date	Autosampler	Method Performed
Cond./Salinity/ TDS meter	HACH 30500006215	Sension 5			No	Conductivity, Salinity, TDS
pH/ ORP meter	HACH 050400020239	Sension 1	2005	2005	No	pH, Oxidation reduction
pH/ ORP meter	HACH 050400022762	Sension 1	2005	2005	No	pH, Oxidation reduction
Cond./Salinity/ TDS meter	HACH 050300013668	Sension 5	2005	2005	No	Conductivity, Salinity, TDS
Cond./Salinity/ TDS meter	YSI 93L12159	33			No	Conductivity, Salinity, TDS
Turbidity meter	LaMotte ME 10036	<b>2</b> 020e	2005	2005	No	Turbidity
Turbidity meter	LaMotie ME 10117	2020e	2005	2005	No	Turbidity
Cond./Salinity/ TDS meter	HACH 050506C50148	Sension 5	2005	2005	No	Conductivity, Salinity, TDS
DO meter	HACH 050500C60212	Sension 6	2005	2005	No	Dissolved oxygen
DO meter	HACH 050500C60066	Sension 6	2005	2005	No	Dissolved oxygen
pH/ ORP meter	HACH 050600C10445	Sension 1	2005	2005	No	pH, Oxidation reduction
pH/ ORP meter	HACH 4030004162	Sension 1	2005	2005	No	pH, Oxidation reduction
DO meter	Hach 040800001267		2006	2006	No	Dissolved Oxygen
Conductivity meter	Hach 050100002708		2006	2006	No	Conductivity
DO meter	Hach 040700001191		2006	2006	No	Dissolved Oxygen
pH/ mV meter	Hach 040200003831		2006	2006	No	pH, mV
Conductivity meter	Hach 050100002707		2006	2006	No	Conductivity
DO meter	Hach 030500007618		2006	2006	No	Dissolved Oxygen
pH/ mV	Hach 041200004666		2006	2006	No	pH, mV
Turbidity meter	LaMotte 4969-1604		2006	2006	No	Turbidity
Turbidity meter	LaMotte 4943-1604		2006	2006	No	Turbidity
Turbidity meter	LaMotte 1909-2900		2006	2006	No	Turbidity
pH/mV meter	Hach 041200002902		2006	2006	No	pH, mV
pH/mV meter E-019	Hach 41200002933	Sension 1	2006	2006	No	pH, mV
Conductivity meter E-027	Hach 050500C50193	Sension 5	2006	2006	No	Conductivity
pH meter E-028	Hach 040800010007	Sension 1	2006	2006	No	pH meter
pH/mV meter M-039	Hach 0804C410063	Sension 1				pH/ORP
pH/mV meter M-034	Hach 06070C710134	Sension 1	Oct06	Oct06	No	pH/ORP
Conductivity meter M-028	Hach 050500C50288	Sension 5	Aug05	Aug05	No	Conductivity

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	Table 21	-1. Example: La	boratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purcha <b>se</b> Date	Install Date	Autosampler	Method Performed
DO meter	Hach	Sension 6	Nov06	Nov06	No	DO
M-032 oH/mV meter	05070C360249 Hach	Sension 1	Oct07	Oct07	No	pH/ORP
N-036 pH/mV meter	07080C710259 Hach	Sension 1	Aug05	Aug05	No	pH/ORP
/I-030 DH/mV meter	050600C10468 Hach	Sension 1	Mar08	Mar08	No	pH/ORP
M-037 DO meter	08020c110145 Hach	Sension 6	2008	2008	No	DO
E-030 oH	07120C260018 Thermo Orion	Model 230			No	pH
-031 H/ORP	018168 Hach	Sension1	2008	2008	No	pH/ORP
E-029	07070C610178 YSI	55/25 FT			No	DO
E-032	01F0708AA Thermo Orion	Model 230A			No	pH
E-033	017788 Thermo Orion	Model 230A			No	pH
E-034 Chlorine meter	017630 Hach	Pocket	2006	2006	No	330.5, SM 18 <sup>th</sup> 4500 CI G
CL-007 Chlorine meter	040200011290 Hach	Colorimeter II Pocket	2006	2006	No	330.5, SM 18 <sup>th</sup> 4500 CI G
CL-002	020100174404	Colorimeter Pocket	2000	2006	No	330.5, SM 18 <sup>th</sup> 4500 CI G
Chlorine meter CL-003	Hach 040200011345	Colorimeter II	1	2006	No	330.5, SM 18 <sup>th</sup> 4500 Cl G
Chlorine meter CL-004	Hach 9612001 <b>02</b> 549	Pocket Colorimeter	2006	2006		330.3, 310 10 4300 01 0
Chlorine meter CL-006	Hach 0304000 <b>34</b> 505	Pocket Colorime <b>ter</b>	2005			
Chlorine meter CL-005	Hach 020100174252	Pocket Colorim <b>eter</b>	2006			
Chlorine meter CL-008	Hach 4796-4900	Colorimeter 1200				
Colorimeter M-040	Hach 041050031426	48450-60 DR/850			No	
Vater level meter	Solonist S/N 37993		Jan05	Feb05	No	
Water level meter	Solonist S/N 37995		Jan05	Feb05	No	
Water level meter	Solonist S/N 42807		Jan06	Jan06	No	
Water level meter	Fisher				No	
PID meter	RAE Systems S/N 110-010953	PGM-7600	May05	May05	No	
PID meter	RAE Systems S/N 110-010984	Mini RAE 2000	May05	May05	No	
PID meter	RAE Systems S/N 110-01094	Mini Rae 2000	May05	May05	No	
PID meter	RAE Systems S/N 103958	Plus Classic	Jan05	Jan05	No	
PID meter	PE Photovac	2020			No	
Comp sampler	S/N DQGD302 ISCO	6037040 <b>01-3700</b>	May05	May05	Yes	
Comp sampler	S/N 205C01376 ISCO S/N 205C01380	6037040 <b>01-3700</b>	May05	May05	Yes	

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	Table 21	-1. Example: La	boratory l	nstrumentati	on List	
Instrument Type	Manuf <b>actur</b> er	Model	Purchase Date	Install Date	Autosampler	Method Performed
Comp sampler	ISCO S/N 204G00984	3700			Yes	
Comp sampler	ISCO S/N 05248-001	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Comp sampler	ISCO	2700			Yes	
Submersible pump	Grundfos S/N 05141-8349	MP1 / 1A106003	May05	May05	No	
Submersible pump	Grundfos S/N 05141-8361	MP1 / 1A106003	May05	May05	No	
Submersible pump	Grundfos S/N 0621-0014	A1A106003P1	Jul06	Jul06	No	
Submersible pump	Grundfos S/N 06029591				No	
Submersible pump	Grundfos S/N 98490294				No	
Submersible pump	Grundfos				No	
Submersible pump	Grundfos				No	
Submersible pump	Grundfos				No	
Submersible pump	Proactive S/N 1371	SS Monsoon	July06	Jul06	No	
Pump control box	Grundfos S/N H0412210120	91126028	May05	May05	No	
Pump control box	Grundfos S/N H0412210120	91126028	May05	May05	No	
Pump control box	Grundfos S/N P1940304254		May05	May05	No	
Pump control box	Grundfos S/N 203831		May05	May05	No	
Pump control box	Grundfos S/N H0303130012		May05	May05	No	
Pump control box	Grundfos S/N 9517		May05	May05	No	
Pump control box	Grundfos		May05	May05	No	
Pump control box	ProActive	Low-flow with power booster	Jul06	Jul06	No	
Trash pump	North Star S/N E06	10633	2007	2007	No	
Generator	Honda S/N EB-3000C	EZGP-1145763	May05	May05	No	
Generator	Honda S/N EB-3000C	EZGP-1151238	Jun05	Jun05	No	
Generator	Honda S/N EZGL1002930	EB-3000C	2005	2005	No	
Generator	Honda				No	
Generator	Honda				No	
Control Pack	QED S/N MP15-1300	MP-15	May05	May05	No	
Control Pack	QED S/N MP15-1297	MP-15	May05	May05	No	

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Nathed Performed						
Instrument Type	Manufacturer	Model	Purcha <b>se</b> Date	Install Date	Autosampler	Method Performed
Control Pack	QED S/N MP15-1298	MP-15	May05	May05	No	
Control Pack	QED S/N MP15-1299	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05	No	
Control Pack	QED	MP-15	May05	May05 May05	No	
Bladder Pump	QED S/N 10993	MP-SPK-4P	May05			
Bladder Pump	QED S/N 10997	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 10995	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 10996	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 11191	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED S/N 11192	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 11512	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 10948	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED 10949	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED	MP-SPK-4P	May05	May05	No	
Bladder Pump	QED	MP-SPK-4P	-		No	
Bladder Pump	QED	MP-SPK-4P			No	
Peristaltic Pump	Solonist S/N 002562	410			No	
Peristaltic Pump	Solonist S/N 002071	410			No	
Peristaltic Pump	Solonist S/N 001979	410			No	
Peristaltic Pump	Solonist S/N 002642	410			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150 portable pump			No	
Peristaltic Pump	ISCO	Accuwell 150			No	
Peristaltic Pump	ISCO	portable pump Accuwell 150			No	
Peristaltic Pump	ISCO	portable pump Accuwell 150			No	
Peristaltic Pump	ISCO	portable pump Accuwell 150			No	
Centrifugal Pump	Teel	portable pump 2P110B			No	

	Table 21-	1. Example:	Laboratory I	nstrumentati	on List	
Instrument Type	Manufacturer	Model	Purchase Date	Install Date	Autosampler	Method Performed
Centrifugal Pump	Teel S/N 0036	2P110B			No	
Centrifugal Pump	Tecl S/N 0034	2P110B			No	
Centrifugal Pump	Teel S/N 1962	2P110B			No	
Centrifugal Pump	Teel	2P110B			No	
Compressor	Coleman / Honda S/N D02812339	CT5090412	Jun05	Jun05	No	
Compressor	Honda/Campbell Hausfeld S/N VT697203AJ				No	
Multi-probe meter YSI-1	YSI S/N 06F1362AC	556 MPS	Jul06	Jul06	No	
GPS	Ashtech 10564	110454-01			No	
Oil/Water Interface probe	Testwell					
Oil/Water Interface probe	Testwell					
Oil/Water interface Probe	Solonist 122-008699-1	122	Sept07	Sept07	No	
Oil/Water interface probe	Solonist S/N 122 007364-1		Aug06	Aug06	No	

Instrument	Procedure	Frequency
		Daily
AA (Quality Europe)	Clean lens and furnace head	As required
(Graphite Furnace)	Replace windows	Daily
	Check or change cuvette	Daily
	Check & drain compressor drain	Daily
	Clean atomizer cell/furnace hood Nebulizer cleaned/dried	Weekly or as required
	Check/change marble stones	Weekly
	Clean filters	Weekly
	Change graphite tube/platform	As required
	Empty waste container	Daily
	Remove carbon tube and check wear	Daily
	Check sample introduction probe	Daily
	· · · · · · · · · · · · · · · · · · ·	
Leeman Mercury	Check tubing for wear	Daily
Analyzer	Fill rinse tank with 10% HCl	Daily As needed
	Change dryer tube	Daily
	Fill reductant bottle with 10% Stannous Chloride	
ICP	Check pump tubing	Daily
	Check liquid argon supply	Daily
	Check fluid level in waste container	Daily
	Check filters	Weekly
	Clean or replace filters	As required
	Check torch	Daily
	Check sample spray chamber for debris	Monthly
	Clean and align nebulizer	Monthly
	Check entrance slit for debris	Monthly
	Change printer ribbon	As required As required
	Replace pump tubing	
ICP MS	Change pump tubing	Weekly or As required
	Clean torch	Weekly or As required
	Check / clean nebulizer	Weekly or As required
	Clean cones	Weekly or As required
	Check air filters	Weekly or As required
	Check multiplier voltages & do cross calibration	Weekly or As required
	Replace sample uptake tubing	Weekly or As required
	Check rotary pump oil	Weekly or As required
	Check oil mist filters	Monthly
	Check chiller water level	Monthly
UV-Vis	Clean ambient flow cell	As required
Spectrophotometer	Precision check/alignment of flow cell	As required
<b>Opodiop</b> hotomotor	Wavelength verification check	Semi-annually
Auto Apolymore	5	Daily
Auto Analyzers	Clean sampler Check all tubing	Daily
	Clean inside of colorimeter	Daily
	Clean pump well and pump rollers	Quarterly
	Clean wash fluid receptacle	Weekly
	Oil rollers/chains/side rails	Weekly
	Clean optics and cells	Quarterly

Table 21-2. Exam	mple: Schedule of Routine Maintenance	
Instrument	Procedure	Frequency
Hewlett Packard/Agilent GC/MS	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment	As required Monthly Annually As required As required As required
	COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required As required As required As required As required
Gas Chromalograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Glass wool replacement Check system for gas leaks with SNOOP Check for loose/frayed wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Daily Daily Daily As required As required

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Table 21-2. Exa	mple: Schedule of Routine Maintenance							
Instrument	Procedure	Frequency						
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required						
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required						
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually						
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required						
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly						
Centrifuge	Check brushes and bearings	Every 6 months or as needed						
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed						

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#### SECTION 22

#### MEASUREMENT TRACEABILITY (NELAC 5.5.6)

## 22.1 OVERVIEW

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 21.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware is suspect, the accuracy of the glassware will be assessed prior to use.

#### 22.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

#### 22.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for

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use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

# 22.4 DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND REFERENCE MATERIALS

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained *i*n the applicable analytical departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

- **22.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.
- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date

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- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained (either electronically or hard-copy) for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**22.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (Specify from LIMS or logbook)
- Special Health/Safety warnings if applicable

**22.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

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## SECTION 23

# SAMPLING (NELAC 5.5.7)

#### 23.1 OVERVIEW

The laboratory provides the following sampling and field services. :

- Groundwater Sampling
- Wastewater Sampling
- Potable Sampling
- Waste Sampling
- Soil and Sediment Sampling
- Flow Monitoring
- Field Parameter Analysis
- Cleaning and Decontamination of Field Equipment

#### 23.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

#### 23.2.1 Preservatives

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid Reagent ACS (Certified VOA Free) or equivalent
- Methanol Purge and Trap grade
- Nitric Acid Instra-Analyzed or equivalent
- Sodium Hydroxide Instra-Analyzed or equivalent
- Sulfuric Acid Instra-Analyzed or equivalent
- Sodium Thiosulfate ACS Grade or equivalent

#### 23.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day

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of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

## 23.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the method SOPs are derived from the source documents for the methods. If method required holding times (as specified in the method SOPs) or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

# 23.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located SOP No. ED-GEN-007 (Subsampling).

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# SECTION 24

## HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

# 24.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 24-1.

#### 24.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 24-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

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The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in the lab job folder.

# 24.1.2 Legal / Evidentiary Chain-of-Custody

The laboratory may, upon special request, adhere to legal/evidentiary chain of custody requirements. If TestAmerica agrees to such procedures the samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal (Figure 24-2), retain the shipping record with the COC, and initiate an internal COC (Figure 24-3) for laboratory use by analysts and a sample disposal record (Figure 24-4).

# 24.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

# 24.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented via the Sample Receipt application within TALS (the laboratory LIMS) and brought to the immediate attention of the appropriate Project Manager who will, in turn, contact the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

#### 24.2.1.1 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 24-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis and necessary QC;
- samples must be preserved according to the requirements of the requested analytical method;

- sample holding times must be adhered to;
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

- **24.2.1.2** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.
- **24.2.1.3** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:
  - Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
  - Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. ED-SPM-001.

#### 24.3 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Sample containers designated for metals only analysis are stored un-refrigerated. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every week.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers, are returned to the secure sample control area. All samples are kept in the refrigerators for 30 days after delivery of the final report to the client, which meets or exceeds most sample holding times. After 30 days the samples are disposed of or, upon client request moved to an un-refrigerated sample archive area where they are stored for an additional time period agreed upon with the client.

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Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

## 24.4 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only.

Procedures for the handling and storage of hazardous samples is addressed in the TestAmerica Corporate Safety Manual (Test America Document No. CW-E-M-001) and in TestAmerica Edison SOP No. ED-SPM-001 (Sample Receipt, Login, Identification, And Storage).

Procedures for the acceptance and handling of USDA regulated domestic and foreign soils are detailed in TestAmerica SOP No. ED-SPM-006 (Procedure for Acceptance and Handling of Regulated Domestic and Foreign Soil).

# 24.5 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

# 24.6 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures, TestAmerica Edison SOP No. ED-SPM-007 (Disposal of Samples and Associated Laboratory Waste). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than 2 months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

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Figure 24-1.

Chain of Custody (COC)

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Figure 24-2

TestAmerica Edison Sample Acceptance Policy

Samples are considered "compromised" if the following conditions are observed upon sample receipt:

- Cooler and/or samples are received outside of temperature specification.
- Samples are received broken or leaking.
- Samples are received beyond holding time.
- Samples are received without appropriate preservation.
- Samples are received in inappropriate containers.
- COC does not match samples received.
- COC is not properly completed or not received.
- Breakage of any Custody Seal (when present)
- Apparent tampering with cooler and/or samples.
- Headspace in volatiles samples >6mm.
- Seepage of extraneous water or materials into samples.
- Inadequate sample volume.
- Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it is documented on the hardcopy COC and within the Sample Receipt application in TALS (the laboratory LIMS) and the client is contacted for instructions. If the client decides to proceed with the analysis, the project report will clearly indicate any of the above conditions and the resolution.

# SECTION 25

## ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

## 25.1 OVERVIEW

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

# 25.2 CONTROLS

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation and drying. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

# 25.3 NEGATIVE CONTROLS

Table 25-1. Example – Negative Con	trols
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Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.
	The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

# Table 25-1. Example – Negative Controls

Control Type	Details
Trip Blank <sup>1</sup>	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks <sup>1</sup>	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks <sup>1</sup>	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

# 25.4 POSITIVE CONTROLS

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

# 25.4.1 Method Performance Control - Laboratory Control Sample (LCS)

- **25.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.
- **25.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through

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all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

- 25.4.1.3 Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- **25.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- 25.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.
  - **25.**4.1.5.1 For methods that have 1-10 target analytes, spike all components.
  - 25.4.1.5.2 For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
  - 25.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.
  - 25.4.1.5.4 Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
  - 25.4.1.5.5 Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

# 25.5 SAMPLE MATRIX CONTROLS

# Table 25-2. Example: Sample Matrix Control

Control Type	(Resolved and a field on a value of a many standard of a more standard of a more standard of a more standard of	Details
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency <sup>1</sup>	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequen <b>cy</b> <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

# 25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

25.6.1 As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

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**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**25.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**25.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking <u>+</u> 3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- **25.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- 25.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- **25.6.3.3** The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- **25.6.3.4** The maximum acceptable recovery limit will be 150%.
- **25.6.3.5** The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- **25.6.3.6** If either the high or low end of the control limit changes by  $\leq 5\%$  from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**25.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

25.6.4.1 The QA department generates Quality Control Limit Summaries in the form of Work Instructions that contain tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Edison This summary includes an effective date, is updated each time new limits are generated and is located in the QAPUBLIC folder on the lab network F: drive. Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into the Laboratory Information Management System

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(LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory.

25.6.5 A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- **25.6.5.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.
- **25.6.5.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.
- **25.6.5.3** Or, for NELAC and Department Of Defense (DOD) work, there are an allowable number of Marginal Exceedances (ME):
  - <11 analytes 0 marginal exceedances are allowed.</p>
  - 11 30 Analytes 1 marginal exceedance is allowed
  - 31-50 Analytes 2 marginal exceedances are allowed
  - 51-70 Analytes 3 marginal exceedances are allowed
  - 71-90 Analytes 4 marginal exceedances are allowed
  - > 90 Analytes 5 marginal exceedances are allowed
  - 25.6.5.3.1 Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (NELAC).
  - 25.6.5.3.2 Marginal exceedances must be random. If the same analyte exceeds the LCS control limit repeatedly, it is an indication of a systematic problem. The source of the error must be located and corrective action taken. The laboratory has a system to monitor marginal exceedances to ensure that they are random.
  - 25.6.5.3.3 Though marginal excedences may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

**25.6.6** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 13.

**25.6.7** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are

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reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

# 25.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

**25.7.1** The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 21), use of certified reference materials (see Section 22) and use of PT samples (see Section 16).

**25.7.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 20.

- **25.7.3** Use of formulae to reduce data is discussed in the method SOPs and in Section 21.
- 25.7.4 Selection of appropriate reagents and standards is included in Section 10 and 22.
- **25.7.5** A discussion on selectivity of the test is included in Section 5.
- **25.7.6** Constant and consistent test conditions are discussed in Section 19.
- **25.7.7** The laboratories sample acceptance policy is included in Section 24.

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# SECTION 26

# REPORTING RESULTS (NELAC 5.5.10)

# 26.1 OVERVIEW

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 20.

# 26.2 <u>TEST REPORTS</u>

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**26.2.1** A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

**26.2.2** Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**26.2.3** A unique identification of the report (e.g. work order number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**26.2.4** A copy of the chain of custody (COC).

• Any COCs involved with Subcontracting are included.

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26.2.5 The name and address of client and a project name/number, if applicable.

26.2.6 Client project manager or other contact

**26.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**26.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**26.2.9** Date reported or date of revision, if applicable.

**26.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

- **26.2.1**1 **Reporting limit.**
- 26.2.12 Method detection limits (if requested)
- **26.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**26.2.14** Sample results.

**26.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**26.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 26.2.4 – Item 3 regarding additional addenda)

**26.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**26.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

**26.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

**26.2.20** When NFLAC accreditation is required, the lab shall certify that the test results meet all requirements of HELAC or provide reasons and/or justification if they do not.

**26.2.21** The laboratory includes a cover letter.

**26.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**26.2.23** When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

**26.2.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**26.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report, or how your lab identifies it). A complete report must be sent once all of the work has been completed.

**26.2.26** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

# 26.2.27 REPORTING LEVEL OR REPORT TYPE

TestAmerica Edison offers several report formats. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 26.2 above.
- Level II (also called 'Results/QA) is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- NJDEP Reduced Deliverables Format which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (Non-USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NJDEP Full Deliverables Format (USEPA CLP Methods) which contains, at minimum, the elements listed in the current NJDEP Technical Requirements for Site Remediation, N.J.A.C. 7:26E.
- NYSDEC ASP 'A' and 'B' Deliverables Format which contain, at minimum, the elements listed in the current New York State Department of Environmental Conservation Analytical Services Protocol.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile or email. All faxed or email reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 26.7.

# 26.2.28 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Edison offers a variety of EDD formats including NJ Hazsite Deliverables, Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

# 26.3 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

**26.3.1** Numeric results with values outside of the calibration range, either high or low are gualified as 'estimated'.

**26.3.2** Where quality system requirements are not met, a statement of compliance/noncompliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

**26.3.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**26.3.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

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When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

# 26.4 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP # CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

# 26.5 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**26.5.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at 732-549-3900 (or for e-mails: please notify us immediately by e-mail or by phone (732-549-3900) and delete this material from any computer).

## 26.6 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

# 26.7 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 13).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the original job number followed by "R". The revised report will have the word "REVISED" next to the report title (i.e., 'Laboratory Results – REVISED'). Any subsequent revisions will be filed on the server under the original job number followed by 'R' and a revision number (ex. R1, R2, R3).

When the report is re-issued, a notation of "REVISED "is placed on the cover/signature page of the report or at the top of the narrative page with a brief explanation of reason for the re-issue.

# 26.8 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

## 26.8.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely <u>no possible</u> impact on the interpretation of the analytical results and there is <u>no possibility</u> of the change being interpreted as misrepresentation by anyone inside or outside of our company.

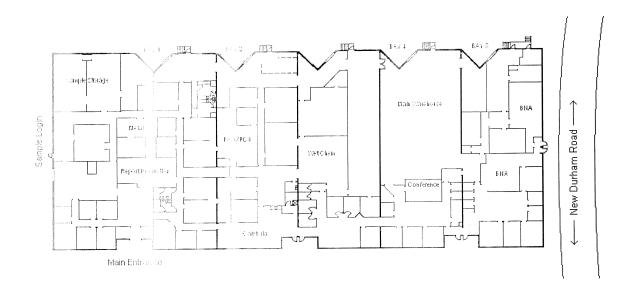
## 26.8.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

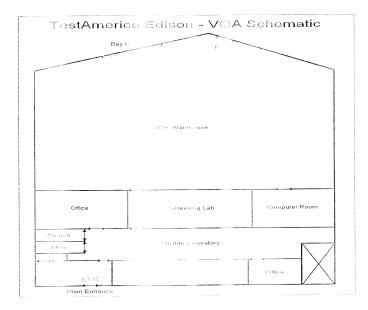
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Appendix 1.

Laboratory Floor Plan



# TestAmerica Edison Facility Schematic



## Appendix 2. Glossary/Acronyms

Glossary:

## Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

## Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

## Accrediting Authority:

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

## Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

## Analyst:

The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

#### Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

#### Blank:

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

#### Blind Sample:

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

#### Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

## Calibration Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

#### Calibration Method:

A defined technical procedure for performing a calibration. (NELAC)

## Calibration Standard:

A substance or reference material used to calibrate an instrument (QAMS)

## Certified Reference Material (CRM):

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

## Chain of Custody:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

#### Clean Air Act:

The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

#### Compromised Samples:

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

## Confidential Business Information (CBI):

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

## Confirmation:

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation Alternate wavelength Derivatization Mass spectral interpretation Alternative detectors or Additional Cleanup procedures

(NELAC)

## Conformance:

An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

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<u>Correction</u>: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

## Corrective Action:

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

#### Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

## Data Reduction:

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

## Deficiency:

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

## Detection Limit:

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

## Document Control:

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

#### Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

## Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

## Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

## External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

## Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

#### Field Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

## Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

#### Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

## Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

## Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

## Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

# Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

#### Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

# Least Squares Regression (1st Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

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In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

## Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

#### Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

## Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure

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to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

#### Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

#### Method Detection Limit:

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

#### Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

#### Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

## Performance Based Measurement System (PBMS):

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

#### Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

#### Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

#### Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

#### Proficiency Testing:

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

#### Proficiency Testing Program:

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

#### Proficiency Test Sample (PT):

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

## Quality Assurance:

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

# Quality Assurance [Project] Plan (QAPP):

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

## Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

## Quality Control Sample:

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

## Quality Manual:

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

## Quality System:

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

#### Quantitation Limits:

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

#### Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

# Reagent Blank (method reagent blank):

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

#### Reference Material:

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

#### Reference Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

## Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

## Report Limit (RL):

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

#### Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

# Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

## Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

<u>Second Order Polynomial Curve (Quadratic)</u>: The 2<sup>nd</sup> order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or r<sup>2</sup>) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r<sup>2</sup> must be greater than or equal to 0.99.

#### Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

## Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

#### Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

## Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

## Standard Operating Procedures (SOPs):

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

## Standardized Reference Material (SRM):

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

#### Surrogate:

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

# Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

#### Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

#### Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

## Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

## Acronyms:

- BS Blank Spike BSD – Blank Spike Duplicate
- CAR Corrective Action Report
- CCV Continuing Calibration Verification
- CF Calibration Factor
- CFR Code of Federal Regulations
- COC Chain of Custody
- CRS Change Request Form
- DOC Demonstration of Capability
- DQO Data Quality Objectives
- DU Duplicate
- DUP Duplicate
- EHS Environment, Health and Safety
- EPA Environmental Protection Agency
- GC Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

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ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy ICV – Initial Calibration Verification IDL – Instrument Detection Limit IH - Industrial Hygiene IS - Internal Standard LCS – Laboratory Control Sample LCSD - Laboratory Control Sample Duplicate LIMS – Laboratory Information Management System MDL – Method Detection Limit MS – Matrix Spike MSD – Matrix Spike Duplicate MSDS - Material Safety Data Sheet NELAC - National Environmental Laboratory Accreditation Conference NELAP - National Environmental Laboratory Accreditation Program PT -- Performance Testing QAM - Quality Assurance Manual QA/QC – Quality Assurance / Quality Control QAPP – Quality Assurance Project Plan RF - Response Factor RPD - Relative Percent Difference RSD - Relative Standard Deviation SD - Standard Deviation SOP: Standard Operating Procedure TAT – Turn-Around-Time VOA - Volatiles VOC – Volatile Organic Compound

# Appendix 3.

# Laboratory Certifications, Accreditations, Validations

*TestAmerica Edison* maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

Organization	Certificate/Lab ID
	Number
New Jersey DEP	12028
Pennsylvania DEP	68-00522
Connecticut DPH	PH-2022
New York DOH	11452
Rhode Island DOH	LAO00132
Delaware DNRC	n/a
USDA Foreign Soils	S-76543
Permit	

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.



# **Cover Page:**

# **Quality Assurance Manual**

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# Title Page:

# Quality Assurance Manual Approval Signatures

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# **REFERENCED CORPORATE SOPS AND POLICIES**

SOP/Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process

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CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

# **REFERENCED LABORATORY SOPs**

SOP Reference	Title
BF-GP-001	Calibration of Autopipettes and Repipetters
BF-GP-002	Support Equipment: Maintenance, Record Keeping and Corrective Actions
BF-GP-005	Sample Homogenization and Subsampling
BF-GP-012	Technical Data Review
BF-GP-013	Manual Integration
BF-GP-015	Record Storage and Retention
BF-GP-018	Strict Internal Chain or Custody
BF-GP-019	Standard Traceability and Preparation
BF-GP-020	Thermometer Calibration
BF-PM-001	Project Information Requirements
BF-PM-003	Bottle Order Set-up
BF-PM-005	Correctness of Analysis
BF-QA-001	Determination of Method Detection Limits
BF-QA-002	Quality Control Limits
BF-QA-003	Procedure for Writing, Reviewing and Revising Controlled Documents
BF-QA-004	Laboratory Personnel Training
BF-QA-005	Preventative and Corrective Action
BF-QA-006	Data Quality Review
BF-SR-001	Cooler Shipping - Bottle Kits and Samples
BF-SR-002	Receipt of Analytical Samples

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#### **SECTION 3**

# INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Buffalo's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999 or 2005 if you're an A2LA lab). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, *Methods for the Determination of Organic Compounds in Drinking Water*, EPA, Revised July 1991.
- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition September 1986, Final Update I, July 1992, Final Update II A, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261. New York State Analytical Services Protocol, July 2005
- <u>Statement of Work for Inorganics & Organics Analysis</u>, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.
- U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.
- U.S. Department of Energy Order 414.1C, Quality Assurance, June 17, 2005.
- U.S. Department of Energy, Quality Systems for Analytical Services, Revision 2.4, October 28, 2008.
- Toxic Substances Control Act (TSCA).

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# 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

# 3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical and biological parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Section 19.0. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director/Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

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# 3.4 MANAGEMENT OF THE MANUAL

# 3.4.1 <u>Review Process</u>

The manual is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & updating procedures (refer to BF-QA-003)

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#### **SECTION 4**

#### ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 <u>OVERVIEW</u>

TestAmerica Buffalo is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Buffalo is presented in Figure 4-1.

#### 4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### 4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Buffalo laboratory.

#### 4.2.2 <u>Laboratory Director</u>

**TestAmerica Buffalo**'s Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

The Laboratory Director has the authority to affect those policies and procedures to ensure that only data of the highest level of excellence are produced. As such, the Laboratory Director is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

Specific responsibilities include, but are not limited to:

- Provides one or more department managers for the appropriate fields of testing. If the Department Manager is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Department Manager to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary NELAC accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.

# Leads the management team, consisting of the QA Manager, the Technical Director, Customer Service Manager, and the Operations Manager as direct reports.

#### 4.2.2 Quality Assurance (QA) Manager

The QA manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA department to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.

- Monitoring and communicating regulatory changes that may affect the laboratory to management.
- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems, data authenticity and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken
- Monitoring standards of performance in quality control and quality assurance.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a subset of all final data reports for internal consistency.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Leads the QA team to enable communication and to distribute duties and responsibilities.
- Ensuring Communication & monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 12.
- Evaluation of the thoroughness and effectiveness of training.
- Compliance with ISO 17025.

# 4.2.3 <u>Technical Director</u>

The Technical Director reports directly to the Laboratory Director and is responsible for assessing the construction and management of the facility design, maintaining environmental conditions, technical and financial evaluation of capital equipment and capital budgeting and asset valuation.

In addition, the Technical Director solves day to day technical issues, provides technical training and guidance to staff, project managers and clients, investigates technical issues identified by operations personnel or QA, and directs evaluation of new methods. Specific responsibilities include but are not limited to:

- Reviewing and approving, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.
- Monitoring the validity of the analyses performed and data generated in the laboratory. This
  activity begins with reviewing and supporting all new business contracts, insuring data
  quality, analyzing internal and external non-conformances to identify root cause issues and
  implementing the resulting corrective and preventive actions, facilitating the data review
  process (training, development, and accountability at the bench), and providing technical
  and troubleshooting expertise on routine and unusual or complex problems.
- Enhancing efficiency and improving quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Compliance with ISO 17025 Standard.

# 4.2.4 **Operations Manager**

The Operations Manager reports to the Laboratory Director and oversees the daily operations of the analytical laboratory, maintaining a working environment that encourages open, constructive problem solving and continuous improvement.

The Operations Manager is responsible for supervision of laboratory staff, setting goals and objectives for the laboratory, ensuring compliance with project/client requirements and ensuring on-time performance, supervises maintenance of equipment and scheduling of repairs. Responsibilities also include implementation of the quality system in the laboratory and ensuring timely compliance with audit and QA corrective actions.

In addition, the Operations Manager works with the Technical Director in evaluating technical equipment, assessing capital budget needs and determining the most efficient instrument utilization. More specifically he:

- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.
- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Technical Director and QA Manager and in compliance with regulatory requirements.
- Works with the Preventive Maintenance Coordinator to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his substitute in the interim.

# 4.2.5 <u>Department Managers</u>

Department Managers report to the Operations Manager. The Department Managers serve as the technical experts on assigned projects, provide technical liaison, assist in resolving any technical issues within the area of their expertise; and implement established policies and procedures to assist the Operations Manager in achieving section goals. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training, and development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Human Resources Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.
- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.

- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager, Technical Director, Operations Manager, and/or Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and longterm needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

#### 4.2.6 <u>Environmental Health & Safety / Hazardous Waste Coordinator</u>

The Health and Safety Coordinator is responsible for the safety and well-being of all employees while at the laboratory. This includes, but is not limited to, administering the Corporate Safety Manual that complies with federal regulations, MSDS training and review, conducting laboratory safety orientation and tours for all new employees, providing instructions on safety equipment, cleaning up laboratory spills, and instructing personnel of laboratory procedures for emergency situations. The Health and Safety Coordinator is on-call 24-hours a day, 7-days a week for all laboratory situations.

The Health and Safety Coordinator responsibilities additionally include waste management of laboratory generated hazardous waste in accordance with appropriate regulations. This includes maintenance of required documentation, such as waste manifests, segregation of waste in accordance with requirements, and training of personnel in proper segregation of waste and preparation of Safety related SOPs. The EHSC maintains overall EH&S program oversight, but may delegate specific day-to-day activities as necessary.

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- Conduct ongoing, necessary safety training and conduct new employee safety orientation.

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- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

# 4.2.7 Laboratory Analysts

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

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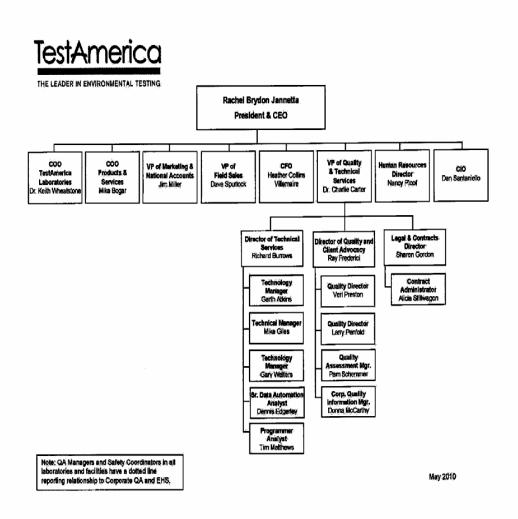
# 4.3 <u>DEPUTIES</u>

The following table defines who assumes the responsibilities of key personnel in their absence:

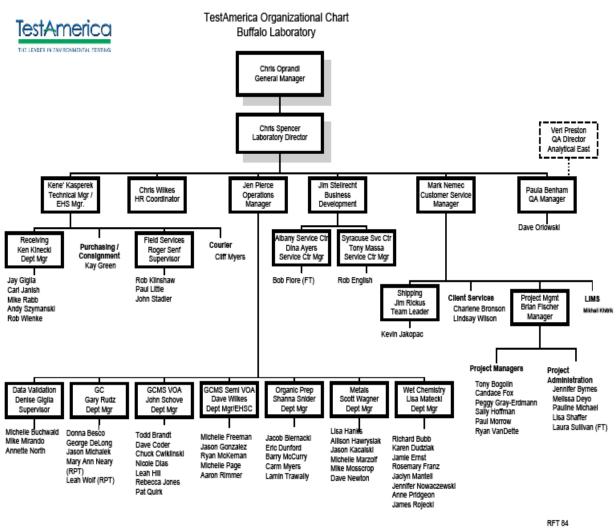
Key Personnel	Deputy	Comment
Laboratory Director	Operations Manager (1) Technical Director (2)	
QA Manager	QA Specialist (1) Operations Manager (2)	
Technical Director	Laboratory Director (1) Operations Manager (2)	
Operations Manager	Department Manager (1) Department Manager (2)	Selected based on availability
Customer Service Manager	Project Mng't Manager (1) Laboratory Director (2)	
Project Management Manager	Customer Srv. Manager (1) Project Manager (2)	(2) Selected based on availability
Project Manager	Project Manager (1) Project Management Asst. (2)	<ul><li>(1) 2° team PM</li><li>(2) Team PMA</li></ul>
Organic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Inorganic Department Manager	Analyst (1) Analyst (2)	Selected based on department, experience and availability
Data Validation / Data Packaging Manager	Data Validation Specialist Data Packaging Specialist	Selected based on department and availability
EHS Coordinator	Safety Officer (1) Sample Mng't Manager (2)	
Sample Management Manager	Sample Custodian (1) EHS Coordinator (2)	
Bottle Preparation / Shipping Manager	Bottle Prep Technician (1) Sample Mng't Manager (2)	

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# Figure 4-1. Corporate and Laboratory Organization Charts



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# **SECTION 5**

# QUALITY SYSTEM (NELAC 5.4.2)

#### 5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

# 5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The 7 elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- Ethics and Compliance Officers (ECOs).
- A training program.
- Self-governance through disciplinary action for violations.
- A confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 16).

- Produce results, which are accurate and include QA/QC information that meets client predefined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.
- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

# 5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents:

- Quality Assurance Manual Each laboratory has a lab specific quality assurance manual.
- <u>Corporate SOPs and Policies</u> Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratories normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- <u>Work Instructions</u> A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- <u>Laboratory SOPs</u> General and Technical
- <u>Corporate Quality Policy Memorandums</u>
- Laboratory QA/QC Policy Memorandums

# 5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies

• Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

# 5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

#### 5.4.1 <u>Precision</u>

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

# 5.4.2 <u>Accuracy</u>

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS.

A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

# 5.4.3 <u>Representativeness</u>

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

# 5.4.4 <u>Comparability</u>

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

# 5.4.5 <u>Completeness</u>

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

# 5.4.6 <u>Selectivity</u>

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

# 5.4.7 <u>Sensitivity</u>

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

# 5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains *Quality Control Limit Data in their LIMS system.* A summary report is generated from LIMS to check the precision and accuracy acceptability limits for performed analyses on request. The summary report is generated and is managed by the laboratory's QA department. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 24.

# 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The procedure for determining the statistical limits may be found in SOP BF-QA-002, Quality Control Limits. The analysts are instructed to use the current limits in the laboratory (dated and approved the QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory through date sensitive tables within the LIMs System. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

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# 5.6.1 QC Charts

The QA Manager periodically evaluates these to determine if adjustments need to be made or for corrective actions to methods (SOP No. BF-QA-002). All findings are documented and kept on file.

# 5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

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# **SECTION 6**

#### DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 <u>OVERVIEW</u>

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. BF-QA-003.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action notices. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

# 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The Quality personnel are responsible for the maintenance of the system.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a Department Manager submits an electronic draft to the QA

Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retain the official document on file. The official document is provided to all applicable operational units. Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years for the majority of procedures and every 1 year for Drinking Water programs. Changes to documents occur when a procedural change warrants.

# 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents". Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. A controlled electronic copy of the current version is maintained on the laboratory IntraNet site and is available to all personnel.

For changes to SOPs, refer to SOP No. BF-QA-003, "Writing, Reviewing and Revising Controlled Documents".

Forms, worksheets, work instructions and information are organized by department in the QA office. Electronic versions are kept in a controlled access electronic folder in the QA department. As revisions are required, a new version number and revision date is assigned and the document placed on the laboratory IntraNet (BufNet) for use.

# 6.4 <u>OBSOLETE DOCUMENTS</u>

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to SOP No. BF-GP-015.

# **SECTION 7**

# SERVICE TO THE CLIENT

#### 7.1 <u>OVERVIEW</u>

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the

contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client and the participating personnel are informed of the changes.

# 7.2 <u>REVIEW SEQUENCE AND KEY PERSONNEL</u>

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- Customer Service Manager
- Operations Manager
- Laboratory and/or Corporate Technical Directors
- Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The Customer Service Manager at the TestAmerica Buffalo facility also maintains copies of these documents.

# 7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Customer Service Manager.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client.

# 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. The PM is the first point of contact for the client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements. Specific information related to project planning may be found in SOP BF-PM-001, Project Information Requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the management staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

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During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during production meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager.

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

# 7.4 SPECIAL SERVICES

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

**Note:** ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representative's cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

# 7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors are available to discuss any technical questions or concerns that the client may have.

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# 7.6 <u>REPORTING</u>

The laboratory works with our clients to produce any special communication reports required by the contract.

# 7.7 <u>CLIENT SURVEYS</u>

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

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# **SECTION 8**

#### SUBCONTRACTING OF TESTS (NELAC 5.4.5)

#### 8.1 <u>OVERVIEW</u>

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process SOP (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the Department of Energy and the USDA, require notification prior to placing such work.

Approval may be documented through reference in a quote / contract or e-mail correspondence.

# 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE) or Customer Service Manager (CSM] becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was

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designated by the client must be maintained with the project file. This documentation can be

- as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica. A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation, where applicable (e.g. on the subcontractors NELAC, A2LA accreditation or State certification.
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work-sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process.

When the potential sub-contract laboratory has not been previously approved, then to begin the process, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought

to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and
- corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report (Form No. CW-F-WI-009).
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories and Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales Personnel.

# 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM, etc.) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontract Laboratory Certification Verification Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilities successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a

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subcontractor facility. If subcontract laboratory data are incorporated into the laboratories EDD (i.e. imported), the report must explicitly indicate which lab produced the data for which methods and samples.

**Note:** The results submitted by TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

# 8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, The QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

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#### Figure 8-1 Subcontracting Laboratory Approval Form (Initial / Renewal)

#### SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date:			
Date: _aboratory: Address:	-		
Address:			

Fax

Contact and e-mail address: Phone: Direct

Requested Item <sup>3</sup>	Date Received	Reviewed/ Accepted	Date
1. Copy of State Certification <sup>1</sup>			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program <sup>3</sup>			
5. QA Manual <sup>3</sup>			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response <sup>1,3</sup>			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit) <sup>3</sup>			
8. Sample Report <sup>3</sup>			
9. SOQ or Summary list of Technical Staff and Qualifications <sup>3</sup>			
10. SOPs for Methods to Be Loadshifted <sup>2,3</sup>			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			

1 - Required when emergency procedures are implemented.2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates. 3 – If the laboratory has NELAC accreditation, <u>Item #s 4 through 10 are not required.</u>

On Site Audit Planned: YES	NO	If yes, Date Complete	d:	By Whom:	
Comments:					
Lab Acceptable for Subcontrac	ting Work	: YES NO	Limitations:		
QA Manager (Signature): Pau	la Benha			Date:	
□ Forwarded to Contract Coor	dinator, b	ру:		Date:	

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# **SECTION 9**

# PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

# 9.1 <u>OVERVIEW</u>

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff.

Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

# 9.2 <u>GLASSWARE</u>

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

#### 9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pretested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001 and TestAmerica Buffalo SOP on Solvent Purity, SOP BF-OP-013.

#### 9.3.1 <u>Purchasing</u>

Chemical reagents, solvents, glassware and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a

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known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP. Purchase requisitions are placed into the J.D. Edwards system by designated departmental personnel. The listing of items available in the J.D. Edwards system has been approved for use by the corporate purchasing staff. Each purchase requisition receives final approval by the laboratory Operations Manager or purchasing coordinator before the order is submitted.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

# 9.3.2 <u>Receiving</u>

It is the responsibility of the purchasing coordinator to receive the shipment. It is the responsibility of the department that ordered the materials to date the material when received. Once the ordered reagents or materials are received, the department that submitted the order compares the information on the label or packaging to the original order to ensure that the purchase meets quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

#### 9.3.3 <u>Specifications</u>

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOP expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in

performing the method and the performance of the dry chemical is found to be satisfactory. The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained along with the calibration raw data for which the reagent was used.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 200 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in the LIMS system, files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

# 9.3.4 <u>Storage</u>

Reagent and chemical storage is important from the aspects of both integrity and safety. Lightsensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. DOC No. CW-E-M-001) and method SOPs or manufacturer instructions.

# 9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical Director and/or the Laboratory Director. If they agree with the request the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

# 9.5 <u>SERVICES</u>

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers, Operations Manager and/or Technical Director.

# 9.6 <u>SUPPLIERS</u>

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurements & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers /vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the

problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

# 9.6.1 <u>New Vendor Procedure</u>

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (available on the intranet site).

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Technical Director are consulted with vendor and product selection that have an impact on quality.

### **SECTION 10**

#### COMPLAINTS (NELAC 5.4.8)

#### 10.1 <u>OVERVIEW</u>

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services, e.g, communications, responsiveness, data, reports, invoicing and other functions expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing with both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following the laboratory SOPs related to Data Quality Review (BF-QA-006) and Corrective Action (BF-QA-005).

#### 10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOPs BF-QA-006 and BF-QA-005.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likely hood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

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The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

## 10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 13. In addition, Corporate Management, Sales and Marketing and Information Technology (IT) may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

## 10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16)

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#### **SECTION 11**

#### CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

#### 11.1 <u>OVERVIEW</u>

When data discrepancies are discovered or deviations and departures from laboratory standard procedures, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the department manager for resolution. The department manager may elect to discuss it with the Technical Director, QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratory's job exception and corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director, Technical Director, Operations Manager or QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with the analytical method requirements and the reason.

## 11.2 RESPONSIBILITIES AND AUTHORITIES

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CA-L-S-001), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances the Laboratory Director, the Technical Director, the Operations Manager or the QA Manager may exceptionally authorize departures from documented

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procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's job exception and corrective action procedures described in Section 12. This information may also need to be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility senior laboratory management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, Technical Director, Operations Manager, QA Manager, Customer Service Manager, Human Resources Manager and Business Development Manager. Suspected misrepresentation issues may also be reported to any member of the corporate staff as identified in Ethics Policy, CA-L-P-001. The data integrity hotline (1-800-736-9407) may also be used. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures <u>must</u> be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

## 11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001 distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

#### 11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system.

On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

#### 11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (i.e., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, Operations Manager, QA Manager, Department Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management and the Customer Service Manager and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

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#### **SECTION 12**

#### CORRECTIVE ACTION (NELAC 5.4.10)

#### 12.1 <u>OVERVIEW</u>

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Report (NCR) also know as Job Exception Reports (JER) and Corrective Action Reports (CAR) (refer to Figure 12-1).

## 12.2 <u>GENERAL</u>

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track client complaints and provide resolution

**12.2.1** <u>Non-Conformance Report (NCR) - (previously known as Job Exception Report</u> and Data Quality Review (DQR) - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non matrix related)
- Isolated reporting / calculation errors
- Client complaints
- Project Management concerns regarding specific analytical results
- Discrepancies in materials / goods received vs. manufacturer packing slips.

**12.2.2** <u>Corrective Action Report (CAR)</u> - is used to document the following types of corrective actions:

• Questionable trends that are found in the monthly review of JERs.

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- Issues found while reviewing JERs that warrant further investigation.
- Questionable trends that are found in the monthly review of DQRs or client complaints
- Internal and External Audit Findings
- Failed or Unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic Reporting / Calculation Errors

## 12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

## 12.3.1 <u>Cause Analysis</u>

- Upon discovery of a non-conformance event, the event must be defined and documented. A NCR or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Department Manager, Operations Manager, Technical Director, or QA Manager (or QA designee) is consulted.

## 12.3.2 <u>Selection and Implementation of Corrective Actions</u>

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCR or CAR is used for this documentation.

## 12.3.3 Root Cause Analysis

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

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Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

## 12.3.4 Monitoring of the Corrective Actions

- The Department Manager, Operations Manager and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers and the Operations Manager are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCR and DQR are entered into a database and each CAR is entered into a spreadsheet for tracking purposes and a monthly summary of all corrective actions is printed out for review to aid in ensuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCR and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the outof-control situation and problems encountered in solving the situation.

## 12.3.5 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.
- Also refer to Section 15.1.4, Special Audits)

## 12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of a NCR or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs. The laboratory may also maintain work instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, work instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly at a minimum by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCR and appropriate corrective action (e.g., reanalysis) is taken and documented.

## 12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, not obliterated (e.g. no white-out), and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

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# Figure 12-1. Example – Corrective Action Notice



October 27, 2010

#### **CORRECTIVE ACTION NOTICE**

CAN #\_\_\_\_\_

Date Issued:	Issued By:
Date Required:	Responsible:
Source of Issue:	

Explanation of Issue: Write a Problem Statement - State Problem - Outline events - Identify people involved	
- Identify missed opportunities	
Investigation Summary: Establish interim containment actions - short term preventive measures Define causal factors & analyze for root cause - Describe investigation (may attach notes, charts, graphs) - Procedures - Training - Quality Control - Communication - Management System - Work Direction	
Root Cause:	
Define causal factors & analyze for root cause - Describe investigation	
(may attach notes, charts, graphs)	
- Procedures - Training - Quality Control	

- Communication - Management System - Work Direction	
Define Root Cause - Summarize findings from problem	
solving	
Impact on Client Data:	
List work orders, batches	
affected and how	
- reanalysis	
<ul> <li>client notification</li> </ul>	
- revised reports	
- data recall	
Corrective Action or Resolution:	
Select Permanent Corrective Actions	
- Alternatives, costs, value added	
- Effective solution - resolve to	
completion	
- within your control	
- measurable	
Timetable for Action:	
Implement permanent corrective action	
<ul> <li>modify procedures</li> </ul>	
- train personnel	
- monitor (plan, do, check, act)	
- adjust if necessary	
Means to Document Corrective	
Action:	
Logbooks, SOPs, Checklists,	
Spreadsheets, Training	
Completed By:	Date:
Approved By:	Date:
Follow-Up Schedule:	
Define interval of follow-up	
State responsible party	
Set reminders	
- Outlook reminder	
- other reminder tools	
Follow-Up Comments:	
Sustained corrective action	
Retain follow-up frequency	

Retain follow-up frequency Not sustained - new investigation - new corrective action	
Follow-Up By:	Date:
Approved By:	Date:

## Table 12-1.

## Example – General Corrective Action Procedures

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action	
Initial Instrument Blank <i>(Analyst)</i>	<ul> <li>Instrument response &lt; MDL.</li> </ul>	<ul> <li>Prepare another blank.</li> <li>If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc</li> </ul>	
Initial Calibration Standards (Analyst, Department Manager)	<ul> <li>Correlation coefficient &gt; 0.99 or standard concentration value.</li> <li>% Recovery within acceptance range.</li> <li>See details in Method SOP.</li> </ul>	<ul> <li>Reanalyze standards.</li> <li>If still unacceptable, remake standards and recalibrate instrument.</li> </ul>	
Independent Calibration Verification (Second Source) (Analyst, Department Manager)	- % Recovery within control limits.	<ul> <li>Remake and reanalyze standard.</li> <li>If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.</li> </ul>	
Continuing Calibration Standards (Analyst, Data Reviewer)	% Recovery within control limits.	<ul> <li>Reanalyze standard.</li> <li>If still unacceptable, then recalibrate and rerun affected samples.</li> </ul>	
Matrix Spike / Matrix Spike Duplicate (MS/MSD) (Analyst, Data Reviewer)	- % Recovery within limits documented in LIMs.	<ul> <li>If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS.</li> <li>If the LCS is within acceptable limits the batch is acceptable.</li> <li>The results of the duplicates, matrix spikes and the LCS are reported with the data set.</li> </ul>	
Laboratory Control Sample (LCS) (Analyst, Data Reviewer)	- % Recovery within limits specified in LIMs.	<ul> <li>Batch must be re-prepared and re- analyzed.</li> <li>Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags.</li> </ul>	

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QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action	
Surrogates (Analyst, Data Reviewer)	<ul> <li>% Recovery within limits of method or within three standard deviations of the historical mean.</li> </ul>	- Individual sample must be repeated. Place comment in LIMS.	
Method Blank (MB) (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	<ul> <li>Reanalyze blank.</li> <li>If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.</li> </ul>	
Proficiency Testing (PT) Samples (QA Manager, Department Manager)	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.	
Internal / External Audits (QA Manager, Department Manager, Operations Manager, Technical Director, Laboratory Director)	- Defined in Quality System documentation such as SOPs, QAM, etc	- Non-conformances must be investigated through CAR system and necessary corrections must be made.	
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.	
Client Complaints (Project Managers, Lab Director, Sales and Marketing, QA Manager)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow- up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).	

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QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director, Operations Manager Department Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation (EH&S Coordinator, Lab Director, Operations Manager, Department Manager)	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected through EH&S office.

#### Note:

1. Except as noted below for certain compounds, the method blank should be below the reporting limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates provided they appear in similar levels in the reagent blank and samples. This allowance presumes that the reporting limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur. For benzene and ethylene dibromide (EDB) and the other analytes for which regulatory limits are extremely close to the detection limit, the method blank must be below the method detection limit.

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#### SECTION 13.0

#### PREVENTIVE ACTION (NELAC 5.4.11)

#### 13.1 <u>OVERVIEW</u>

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's Corrective Action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system:

- <u>Identification</u> of an opportunity for preventive action.
- <u>Process</u> for the preventive action.
- <u>Define the measurements of the effectiveness of the process once undertaken.</u>
- <u>Execution</u> of the preventive action.
- Evaluation of the plan using the defined measurements.
- <u>Verification</u> of the effectiveness of the preventive action.
- <u>Close-Out</u> by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review

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**13.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 17). A highly detailed recap is not required; a simple recount of success and failure within the preventive action program will provide management a measure for evaluation.

## 13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these procedures, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of changes covered under this system include: Facility Changes, Major Accreditation Changes, Addition or Deletion to Division's Capabilities or Instrumentation, <u>Key</u> Personnel Changes, Laboratory Information Management System (LIMS) changes.

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#### SECTION 14.0

#### CONTROL OF RECORDS (NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued. TestAmerica Buffalo SOP BF-GP-015, Record Storage and Retention specify additional storage, archiving and retention procedures.

### 14.1 <u>OVERVIEW</u>

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database which is backed up as past of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Hardcopy technical records are maintained by the Data Deliverables Manager while electronic technical records are maintained by the IT Administrator.

	Record Types <sup>1</sup> :	Retention Time:
Technical Records	<ul> <li>Raw Data</li> <li>Logbooks<sup>2</sup></li> <li>Standards</li> <li>Certificates</li> <li>Analytical Records</li> <li>Lab Reports</li> </ul>	5 Years from analytical report issue*
Official Documents	<ul> <li>Quality Assurance Manual (QAM)</li> <li>Work Instructions</li> <li>Policies</li> <li>Policy Memorandums</li> <li>SOPs</li> <li>Manuals</li> </ul>	5 Years from document retirement date*
QA Records	<ul> <li>Internal &amp; External Audits/Responses</li> <li>Certifications</li> <li>Corrective/Preventive Actions</li> <li>Management Reviews</li> <li>Method &amp; Software Validation / Verification Data</li> <li>Data Investigation</li> </ul>	5 Years from archival* <u><b>Data Investigation:</b></u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)

#### Table 14-1. Record Index<sup>1</sup>

	Record Types <sup>1</sup> :	Retention Time:
Project Records	<ul> <li>Sample Receipt &amp; COC</li> <li>Documentation</li> <li>Contracts and Amendments</li> <li>Correspondence</li> <li>QAPP</li> <li>SAP</li> <li>Telephone Logbooks</li> <li>Lab Reports</li> </ul>	5 Years from analytical report issue*
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely
	Administrative Policies Technical Training Records	7 years

<sup>1</sup>Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. Retention of records is maintained on-site at the laboratory for at least 3 months after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless other wise specified by a client or regulatory requirement. All records shall be protected against fire, theft, loss, environmental deterioration and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to the data is limited to laboratory and company employees.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

## 14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data. Specific Information related to archival of data for greater than 5 years may be found in TestAmerica Buffalo SOP BF-GP-015.

#### Table 14-2. Special Record Retention Requirements

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	10 years (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
FIFRA – 40 CFR Part 160	Retain for life of research or marketing permit for pesticides regulated by EPA
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
Alaska	10 years
Louisiana – All	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	5 years
NY Potable Water NYCRR Part 55-2	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup>Note: Extended retention requirements are noted with the archive documents or addressed in TestAmerica Buffalo facility-specific records retention procedure BF-GP-015.

**14.1.3** All records are held secure and in confidence. Records maintained at the laboratory are located in the locked on-site storage room. Records archived off-site are stored in a secure location. Access to the off-site storage facility is controlled and logs are maintained for the documented removal/return of records

**14.1.4** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. TestAmerica Buffalo SOP BF-GP-015 also contains specific information for archival of scanned data.

**14.1.5** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (records

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stored off site should be accessible within 2 business days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the chain of custody is stored with the project file and the Job Number analytical service request form (ASRF) generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Instrument data is stored sequentially by instrument. Calibration data for a given sequence are maintained in the order of the analysis. Sample data are stored on a job number basis in the project file or as part of the daily batch or sequence. Run logs are maintained for each instrument or method; a copy of each day's run log or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks, bench sheets or excel spreadsheets are used to record and file data. Standard and reagent information is recorded in logbooks or on the raw data for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in TestAmerica SOP BF-GP-015.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

## 14.2 TECHNICAL AND ANALYTICAL RECORDS

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the sampling, performance of each analysis and reviewing of results.

**14.2.2** Observations, data and calculations are recorded real-time.

**14.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 13 and 20. Changes to electronic records in LIMS or instrument data are recorded in audit trails. The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in the method specific SOPs, in the instrument method detail records or the instrument maintenance logs where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods, ID codes, volumes, weights, instrument printouts, meter readings, temperatures, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and

• Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

## 14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a
  description of the specific computational steps used to translate parametric observations into
  a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

#### 14.3.1 Sample Handling Records

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- Procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

## 14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

## 14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

**14.5.1** All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

- **14.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.
- **14.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.
- **14.5.4** The laboratory has a record management system (also known as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per instrument or analysis basis, and are numbered sequentially as they are issued. No instrument or analysis has more than one active notebook at a time, so all data are recorded sequentially within a series of sequential notebooks. Bench sheets and raw data sequence files are filed sequentially by date. Standard and reagent information is maintained in LIMS and logbooks which are maintained on a departmental basis and are numbered sequentially as they are issued or as they are archived by QA.
- **14.5.5** Records are considered archived when noted as such in the records management system (also known as document control). Access to archived hard-copy information is documented with an access log and in/out records is used to note data that is removed and returned.

## 14.5.6 <u>Transfer of Ownership</u>

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

#### 14.5.7 <u>Records Disposal</u>

**14.5.7.1** Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

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- **14.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.
- **14.5.7.3** If a third party records Management Company is hired to dispose of records, a "Certificate of Destruction" is required.

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#### **SECTION 15**

## AUDITS (NELAC 5.4.13)

#### 15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits *	QA Department or Designee	All methods within a 2-year period (50% annually)
SOP Method Compliance Audits *	Dept. Manager or designee	All methods within a 2-year period (50% annually)
Special	QA Department or Designee	Surveillance or spot checks performed as needed to monitor specific issues
Performance Testing	Coordinated by Corporate QA	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

#### Table 15-1. Types of Internal Audits and Frequency

\* = all methods receive a QA Technical Audit or an SOP Method Compliance Audit annually.

#### 15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given area. The area audits may be done on a rotating schedule throughout the year to ensure

adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

## 15.1.2 **QA Technical Audits**

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, Chrom AuditMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

## 15.1.3 <u>SOP Method Compliance</u>

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

## 15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

## 15.1.5 <u>Performance Testing</u>

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Nonpotable Water, Soil, Air.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

## 15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

## 15.2.1 <u>Confidential Business Information (CBI) Considerations</u>

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

## 15.3 AUDIT FINDINGS

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

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### **SECTION 16**

#### MANAGEMENT REVIEWS (NELAC 5.4.14)

#### 16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director for review and comments. The final report shall be submitted to the Operation Manager as well as the appropriate Quality Director and General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Director prepares a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

## 16.2 ANNUAL MANAGEMENT REVIEW

The senior lab management team (Laboratory Director, Technical Director, Operations Manager, Customer Service Manager, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals and objectives. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective; therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.

- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes.

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

#### 16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. The TestAmerica Corporate Data Investigation/ Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

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#### **SECTION 17**

#### PERSONNEL (NELAC 5.5.2)

#### 17.1 <u>OVERVIEW</u>

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

## 17.2 <u>EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL</u> <u>PERSONNEL</u>

The laboratory makes every effort to hire analytical staff that possesses a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are

located in the TestAmerica Buffalo Human Resource office (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, pipette, quantitation techniques, etc. are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC)	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience
Technical Directors/Department Managers – <u>General</u>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

## 17.3 <u>TRAINING</u>

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics - Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 20).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- The Human Resource office maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in TestAmerica Buffalo SOP BF-QA-004, Laboratory Personnel Training.

## 17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire followed by technical data integrity training within 30 days, comprehensive

training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy No. CA-L-P-001 and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

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#### **SECTION 18**

## ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

## 18.1 <u>OVERVIEW</u>

TestAmerica Buffalo is a 32,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc. OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for field operations, bottle kit preparation, sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis and administrative functions.

## 18.2 ENVIRONMENT

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may affect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory. Key equipment has been provided with back-up power supply in the event of a power outage.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## 18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

• Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

## 18.4 <u>FLOOR PLAN</u>

A floor plan can be found in Appendix 1.

## 18.5 BUILDING SECURITY

Building pass cards and alarm codes are distributed to all facility employees.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. [The reason for this is that it is important to know who is in the building in case of a safety emergency. The visitors logbook is used to ensure that everyone got out of the building safely.] In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

#### **Company Confidential & Proprietary**

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

#### SECTION 19.0

# TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

# 19.1 <u>OVERVIEW</u>

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

# 19.2 STANDARD OPERATING PROCEDURES (SOPs)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory:

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP) and Laboratory SOP BF-QA-003, Procedure for Writing, Reviewing and Revising Controlled Quality Documents (QAM, SOP, etc)
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

#### 19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from

the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

# 19.4 <u>SELECTION OF METHODS</u>

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists, etc.), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

# 19.4.1 Sources of Methods

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

**19.4.1.1** The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- <u>Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel</u> <u>Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and</u> <u>Gravimetry</u>, EPA-821-R-98-002, February 1999
- <u>Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air</u>, US EPA, January 1996.
- <u>Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act</u>, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. <u>Revised as of July 1, 1995, Appendix</u> <u>A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)</u>
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- <u>Methods for the Determination of Inorganic Substances in Environmental Samples</u>, EPA-600/R-93/100, August 1993.

- <u>Methods for the Determination of Metals in Environmental Samples</u>, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- <u>Methods for the Determination of Organic Compounds in Drinking Water</u>, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. <u>Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series)</u> (EPA 500 Series methods)
- <u>Technical Notes on Drinking Water Methods</u>, EPA-600/R94-173, October 1994
- <u>NIOSH Manual of Analytical Methods</u>, 4<sup>th</sup> ed., August 1994.
- <u>Statement of Work for Inorganics & Organics Analysis</u>, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- <u>Standard Methods for the Examination of Water and Wastewater</u>, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup> / on-line edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- <u>Annual Book of ASTM Standards</u>, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- <u>National Status and Trends Program</u>, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.
- <u>Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January</u> 2005) (DW labs only)
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261
- New York State DEC Analytical Services Protocol, 2005
- <u>New York State DOH Methods Manual</u>

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### 19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not

test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

- **19.4.2.1** A demonstration of capability (BF-QA-004) is performed whenever there is a significant change in instrument type (e.g., new instrumentation), method or personnel.
- **19.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Operations Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.
- **19.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct a method detection limit study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

# 19.4.3 Initial Demonstration of Capability (IDOC) Procedures

Procedures for generation of IDOCs are detailed below and in laboratory SOP BF-QA-004, Laboratory Personnel Training.

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

- **19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.
- **19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).
- **19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.
- **19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.
- **19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.
- **19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:
  - Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
  - Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 20.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (see Figure 19-1) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

# 19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## 19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

#### 19.6.1 <u>Method Validation and Verification Activities for All New Methods</u>

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

#### 19.6.1.1 Determination of Method Selectivity

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

# 19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

#### 19.6.1.3 <u>Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)</u>

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

#### 19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

#### 19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### 19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### 19.6.1.7 Documentation of Method

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### 19.6.1.8 <u>Continued Demonstration of Method Performance</u>

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

# 19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods. whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used.

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. BF-QA-001 for details on the laboratory's MDL process.

## 19.8 INSTRUMENT DETECTION LIMITS (IDL)

**19.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**19.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation. (For CLP procedures, the IDL is determined using the standard deviation of 7 replicate spike analyses on each of 3 non-consecutive days.)

**19.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

# 19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

**19.9.1** Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, CVAA, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified or see section 20.7.9 for other options. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually

**19.9.2** When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory

# 19.10 **RETENTION TIME WINDOWS**

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory's Sops.

# 19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, and specific electrode response factors.

# 19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

**19.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 + -0.5 mg/l.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g. 524.2, 525, etc) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

## 19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within <u>+</u> 1 reporting limit for samples ≤ 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.
- Due to the potential for increased variability, reanalysis may not be applicable to Nonhomogenous, Encore, and Sodium Bisulfate preserved samples. See the Department Supervisor or Laboratory Director/Manager if unsure.

# 19.14 <u>CONTROL OF DATA</u>

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### 19.14.1 <u>Computer and Electronic Data Related Requirements</u>

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the 'TALS Data System' which is a LIMs system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes a SQL server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

#### 19.14.1.1 Maintain the Database Integrity

Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

• LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.

• Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.

# 19.14.1.2 Ensure Information Availability

Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

# 19.14.1.3 Maintain Confidentiality

Ensure data confidentiality through physical access controls, when electronically transmitting data.

# 19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The data review sheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- **19.14.2.1** All raw data must be retained in the project job folder, computer file, and/or run log. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/<u>year</u>). It must be easily identifiable who performed which tasks if multiple people were involved.
- **19.14.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter (μg/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram (μg/kg) for solids. For values greater than 10,000 mg/l,

results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

- **19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, final inorganic results are reported to 2 significant figures for values less than 10 and 3 significant figures for values greater than 10 on the final report. Organic results are generally reported to 1 significant figure for values less than 10 and 2 significant figures for values greater than 10 on the final report. The number of significant figures may be adjusted based on client or project requirements.
- **19.14.2.4** For those methods that do not have an instrument printout, an instrumental output or a calculation spreadsheet upload compatible with the LIMS System, the final results and dilution factors are entered directly into LIMS by the analyst, and the software formats the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- **19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is automatically transferred to the network server and, eventually, to a back-up tape file.

# 19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Technical Director/QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

#### 19.14.4 <u>Review / Verification Procedures</u>

Review procedures are out lined in several laboratory SOPs (e.g. BF-SR-002, "Receipt of Analytical Samples", BF-GP-012, "Technical Data Review", and BF-PM-001, "Project Information Requirements") to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (BF-GP-013, Manual Integration). The general review concepts are discussed below, more specific information can be found in the SOPs.

- **19.14.4.1** The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Project Managers perform review of the chain-of-custody forms and inputted information and approve the input in LIMs to make the samples available to the laboratory departments for batching and processing.
- **19.14.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add any manual data qualifiers or dilution codes if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 10% of all sample data from manual integrations are reviewed. Issues that deem further review include the following:
  - QC data are outside the specified control limits for accuracy and precision
  - Reviewed sample data does not match with reported results
  - Unusual detection limit changes are observed
  - Samples having unusually high results
  - Samples exceeding a known regulatory limit
  - Raw data indicating some type of contamination or poor technique
  - Inconsistent peak integration
  - Transcription errors
  - Results outside of calibration range
  - Results deviate from historical trends (if history available)

- **19.14.4.3** Unacceptable analytical results may require reanalysis of the samples. Any unusual or uncharacteristic circumstances are brought to the attention of the Department Manager. The Department Manager may involve the Project Manager, the Technical Director and/or the QA Manager for further investigation depending on the issue. Corrective action is initiated whenever necessary.
- **19.14.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.
- **19.14.4.5** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.
- **19.14.4.6** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report and creates the invoice. When complete, the report is issued to the client.

# 19.14.5 <u>Manual Integrations</u>

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using SOP CA-Q-S-002 as the guidelines.

- **19.14.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required. Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.
- **19.14.5.2** Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.

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- **19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- **19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

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#### Figure 19-1. Example - Demonstration of Capability Documentation



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DOC Cert. Statement Revision 9 April 13, 2010

# **TESTAMERICA LABORATORIES, INC.**

#### TRAINING & DEMONSTRATION OF CAPABILITY CERTIFICATION STATEMENT

Employee:		Page _	of
Method Number:		Date:	
Parameters or Analytes:			
Initial Demonstration of Capability:			
SOP Number:	Revision #	Date Read	_
Trained By:			
Date training began:	Date traini	ng completed:	
Continued Demonstration of Capability	v: 🗆		
SOP Number:		Revision #	Date Read
I CERTIFY that I have read and understan capability.	nd the SOP identified above	. I have also submitted data asso	becated with the demonstration of
		Employee Signature	- Date
We, the undersigned, CERTIFY that:			
1. The analyst identified above, using the National Environmental Laboratory Accre			
2. The test method(s) was performed by the	he analyst(s) identified on the	nis certification.	
3. A copy of the test method(s) and the lab	boratory-specific Sops are a	vailable for all personnel on-site	
4. The data associated with the demonstra	tion capability are true, acc	urate, complete and self-explanat	ory.
5. All raw data (including a copy of this c facility, and that the associated information			
Jennifer Pierce	Signature		Date
Paula Benham Quality Assurance Manager	Signature		Date

#### **SECTION 20**

#### EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

#### 20.1 <u>OVERVIEW</u>

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory equipment and instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturer's instructions for equipment use are readily accessible to all appropriate laboratory personnel.

#### 20.2 PREVENTIVE MAINTENANCE

**20.2.1** The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**20.2.2** Routine preventive maintenance procedures and frequency, such as lubrication, cleaning, and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

**20.2.3** Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may also be outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

**20.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

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**20.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

**20.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrumentation records.

**20.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

**20.2.5** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out of service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses

**20.2.6** In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

# 20.3 <u>SUPPORT EQUIPMENT</u>

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, field sampling devices, temperature measuring devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance. Laboratory SOPs BF-GP-001, "Calibration of Autopipettes and Repipetters" and BF-GP-002, "Support Equipment: Maintenance, Record Keeping and Corrective Actions of Analytical Balances, Temperature Control Devises and Reagent Water" provide additional detail on the monitoring and record keeping for support equipment.

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#### 20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file.

#### 20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to  $\pm$  0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

#### 20.3.3 Thermometers

All reusable thermometers are calibrated on an annual basis with a NIST-traceable thermometer. Disposable glycol thermometers are discarded upon expiration and replaced with newly purchased thermometers. IR thermometers are verified daily and calibrated annually. Digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside

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service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories) and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the laboratory SOP BF-GP-020, "Thermometer Calibration".

#### 20.3.4 <u>Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators</u>

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between >  $0^{\circ}$ C and  $\leq 6^{\circ}$ C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

#### 20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically at a minimum on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

#### 20.3.6 Field Sampling Devices (Isco Auto Samplers)

Each Auto Sampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly (or if not utilized monthly, immediately prior to its usage) by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder.

Additional calibration and use information is detailed in laboratory SOP BF-FS-006, "Calibration of Field Meter".

#### 20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:** Instruments are calibrated initially and as needed after that and at least annually.

#### 20.4.1 Calibration Standards

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

- **20.4.1.1** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.
- **20.4.1.2** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).
- **20.4.1.3** The other concentrations define the working range of the instrument/method or correspond to the expected range of concentrations found in actual samples that are also within the working range of the instrument/method. Results of samples not bracketed by initial instrument calibration standards (within calibration range to 3 significant figures) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exceptions to these rules are methods where the referenced method does not specify two or more standards.
- **20.4.1.4** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

# 20.4.2 Calibration Verification

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard) to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met i.e., RPD, per NELAC (2003) Standard, Section 5.5.5.10.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

Note: If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

# 20.4.2.1 <u>Verification of Linear and Non-Linear Calibrations</u>

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

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- When the acceptance criteria for the calibration verification are exceeded high, i.e., high bias, and there are associated samples that are non-detects, then those non-detects may be reported. Otherwise, the samples affected by the unacceptable calibration verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted.
- When the acceptance criteria for the calibration verification are exceeded low, i.e., low bias, those sample results may be reported if they exceed a maximum regulatory limit/decision level. Otherwise, the samples affected by the unacceptable verification shall be reanalyzed after a new calibration curve has been established, evaluated and accepted. Alternatively, a reporting limit standard may be analyzed to demonstrate that the laboratory can still support non-detects at their reporting limit.

# 20.5 <u>TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS</u>

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification. See laboratory SOP's BF-MB-005 and BF-MV-007 for guidelines for making tentative identifications

#### Note:

For general reporting if TICs are requested, the ten (10), largest non-target analyte peaks whose area count exceeds 10% of the nearest internal standard will be termed "Tentatively Identified Compounds" (TICs). More or fewer TICs may be identified based on client requirements.

# 20.6 <u>GC/MS TUNING</u>

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

Instrumentation	Serial	Dato In	Autosample	Conditio	Hazelwood	Method(s)
instrumentation	Number	Service	r	n	Floor Plan	Performed
	Number				Location	T CHOIMCU
GC/MS Instrumentation	_			-	Location	
Agilent 5975	CN10833020	2009	Yes	good	T	8260B
Agilent 5975	US80838844	2003	Yes	good	C	8260B
Agilent 5973	US44621446	2005	Yes	good	G	8260B
Agilent 5973	US52420646	2005	Yes	good	J	8260B
Agilent 5973	US41720721	2003	Yes	good	S	8260B
Agilent 5973	US35120354	2004	Yes	good	Ŵ	8270C, 625
Agilent 5973	US41720707	2004	Yes	good	X	8270C, 625
Agilent 5973	US10241053	2004	Yes	good	R	8260B
Agilent 5973	US30965634	2003	Yes	good	V	8270C, 625
Agilent 5973	US03965692	2003	Yes	good	Ŭ	8270C, 625
Agilent 5973	US05060076	2000	Yes	good	F	8260B
Agilent 5973	US05060084	2001	Yes	good	N	8260B
Agilent 5973	US03950346	2001	Yes	good	P	8260B
Agilent 5973	US82321636	2001	Yes	good	Q	8260B
GC Instrumentation	0002021000	2001	100	good	<u> </u>	02002
Agilent 6890 dual uECD	CN10520009	2005	Yes	good	5	8081
Agilent 6890 dual uECD	CN10520010	2005	Yes	good	6	CLP Pest/PCB
Agilent 6890 dual uECD	CN10448015	2005	Yes	good	7	8082
Hewlett Packard 5890II dual ECD	3336A53126	1994	Yes	good	14	8082
Hewlett Packard 5890II dual ECD	3336A63465	1994	Yes	good	15	RSK
Hewlett Packard 5890II dual ECD	3336A53464	1994	Yes	good	16	Screening
Hewlett Packard 5890II dual ECD	3336A53463	1994	Yes	good	17	504, 8011
Hewlett Packard 5890II dual ECD	3336A54409	1994	Yes	good	18	608, 8081
Hewlett Packard 5890II dual ECD	3336A54408	1994	Yes	good	19	8082
Hewlett Packard 5890II FID/FID	3115A34892	1994	Yes	good	2	NIOSH Air
Hewlett Packard 5890II PID/FID	3336A60622	1994	Yes	good	22	8021B
Hewlett Packard 5890II Hall/PID	3235A54089	1994	Yes	good	0	8021B
Hewlett Packard 5890II PID/FID	3336A53465	1994	Yes	good	23	8021B
Hewlett Packard 5890II dual FID	3336A53727	1994	Yes	good	24	8015 DRO
Hewlett Packard 5890II dual ECD	3310A47661	1993	Yes	good	12	8082
Hewlett Packard 5890II dual ECD	3336A53325	1993	Yes	good	13	8151A
Hewlett Packard 5890II PID/FID	3133A37157	1993	Yes	good	8	GRO
Hewlett Packard 5890II dual ECD	3203A42206	1992	Yes	good	9	CLP Pest/PCB
Hewlett Packard 5890II dual FID	3019A28433	1991	Yes	good	4	8015 Modified
Hewlett Packard 5890II Hall/PID	3121A35782	1990	Yes	good	3	8021B
Metals Instrumentation						
Perkin Elmer Elan 9000 ICP-MS	P0230202	2002	Yes	good	PE	6020, 200.8
Leeman PS200 II	HG9045	2000	Yes	good	Hg Lab	7471A, 7470,

# Table 20-1. Laboratory Equipment and Instrumentation – TestAmerica Buffalo

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						245.1
Leeman PS200 II	HG0033	2000	Yes	good	Hg Lab	7471A, 7470, 245.1
Thermo Jarrell ICAP 6000 Duo	ICP- 20094603	2010	Yes	good	TJA	6010B, 200.7
Thermo Jarrell ICAP 6000 Duo	ICP- 20094602	2010	Yes	good	TJA	6010B, 200.7
Water Quality Instrumentation					(Suite 108)	
Konelab Aqua20	SEA032	2009	Yes	good	WQ Lab	350.1, 351.2, 9012
Flash Point Analyzer	Herzog	2007	Yes	good		1010
OI Carbon Analyzer Model 1030	A54TB0578P	2006	Yes	good	41	415.1, 9060
OI Carbon Analyzer Model 1030	E616130020 E	2006	Yes	good	41	415.1, 9060
Thermo ECA 1200 TOX	2006.0373	2006	Yes	good		9020B
Horizon Speed Vap	03-0415	2005	Yes	good	WQ Lab	1664
Konelab 20XT	E3719731	2005	Yes	good	WQ Lab	350.1, 351.2, 9012
Thermo ECA 1200 TOX	2004.901	2004	Yes	good	7	9020B
Dionex Ion Chromatograph #DX- 120	20126	2004	Yes	good	35	VFA
Konelab 20	S5019455	2004	Yes	good	WQ Lab	310.2, 325.1, 375.4
Glastron CN Midi-distillation	2502	2003	n/a	good	5	335.2, 9010A, 9012
Glastron Phenol Midi-distillation	2069	2003	n/a	good	30	420.2, 9066
Glastron Phenol Midi-distillation	2053	2003	n/a	good	31	420.2, 9066
Labtronics BOD Magic - Autoanalyzer	270H3XB531	2004	Yes	good	WQ Lab	405.1
Labtronics BOD Magic - Autoanalyzer	270J2XB669	2003	Yes	good	WQ Lab	405.1
ManTech PC Titrator	MS-OK2-607	2003	Yes	good	WQ Lab	120.1, 150.1, 310.1, 340.2
HACH Spectrophotometer #DR/2500	30200004886	2003	n/a	good	WQ Lab	365.2,410.4, 7196A
Dionex Ion Chromatograph #DX- 120	2060196	2002	Yes	good	29	300.0, 9056
Spectronic Genesis 4001/4	3SGC199091	2000	n/a	good	3	335.2, 9010A
Lachat Quickchem 8000 Autoanalyzer	A83000-1527	2000	Yes	good	27	335.4, 9012, 9066
OI Carbon Analyzer Model 1010 #1	H92170411	1999	Yes	good	1	415.1, 9060
Lachat Quickchem 8000 Autoanalyzer	A83000-1439	1999	Yes	good	9	350.1, 353.2
Dionex Ion Chromatograph #DX- 120	99010157	1999	Yes	good	28	300.0, 9056
Orion Ion Meter #230A	2229	1999	n/a	good	WQ Lab	150.1, 9040A
VWR Ion Meter #2100	1063	1997	n/a	good	WQ Lab	150.1, 9040A
YSI Oxygen Meter #57	93J09826	1995	n/a	good	WQ Lab	405.1
BOD chamber	Revco	1994	n/a	good	6	405.1
Sample Preparation Equipment		-	-			
TurboVap II	TV0529N124 27	2006	n/a	good	39	n/a

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TurboVap II	TV0529N124 28	2006	n/a	good	38	n/a
J2 ACCUPREP GPC	03F-10723	2003	Yes	good	10	3640A
TurboVap II	TV9445N581 6	1996	n/a	good	17	n/a
TurboVap II	TV9427N413 3	1996	n/a	good	18	n/a
TurboVap II	TV944N5819	1996	n/a	good	19	n/a
TurboVap II	TV944N5820	1996	n/a	good	20	n/a
TurboVap II	TV0024N962 3	2000	n/a	good	32	n/a
TurboVap II	TV0022N960 4	2000	n/a	good	25	n/a
TurboVap II	TV0312N115 92	2003	n/a	good	33	n/a
TurboVap II	TV0312N115 91	2003	n/a	good	34	n/a
Organomation Rot-X-Tractor	16902	1999	n/a	good	11	3510C
Organomation Rot-X-Tractor	16907	1999	n/a	good	12	3510C
Organomation Rot-X-Tractor	16913	1999	n/a	good	13	3510C
Heat Systems Sonicator #XL-2020	G1647/C5659	1994	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2665/C5674	1994	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2620/C5660	1994	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2245/C6328	1995	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2621/C6733	1995	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2713/C6732	1995	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G1643/C6837	1995	n/a	good	O-Prep Lab	3550B
Heat Systems Sonicator #XL-2020	G2742/C6842	1995	n/a	good	O-Prep Lab	3550B

Table 20-2.

# Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Change dryer tube Fill reductant bottle with 10% Stannous Chloride	Daily Daily As Needed Daily
ICP & ICP/MS	Check pump tubing Check liquid argon supply Check fluid level in waste container Check re-circulator levels Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Change pump oil Change Cones Change printer cartridge Replace pump tubing	Daily Daily Daily Monthly As required Daily Monthly Monthly Monthly As required As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly
Agilent GC/MS	Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment	Monthly Annually As required As required As required
	COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required As required As required As required As required

Instrument	Procedure	Frequency
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column Check temp. of detector, inlet, column oven Septum replacement Glass wool replacement Check system for gas leaks with SNOOP Check for loose/frayed power wires and insulation Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily Daily via use of known compound retention Daily As required As required W/cylinder change as required As Required As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples and solvents Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Vacuum Pumps/ Air Compressor	Drained Belts checked Lubricated	Weekly Monthly Semi-annually
Centrifuge	Check brushes and bearings	Every 6 months or as needed

# Table 20-3.

# **Periodic Calibration**

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Analytical Balance	Accuracy determined using "S" NIST traceable weights. Minimum of 2 standards bracketing the weight of interest.	Daily, when used	± 0.2%	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service.
	Inspected and calibrated by A2LA accredited person annually.	Annual		
Top Loading Balance	Accuracy determined using "S" NIST traceable. Minimum of 2 standards bracketing the weight of interest.	Daily, when used	± 0.5%	Clean. Replace.
	Inspected and calibrated by A2LA accredited person annually.	Annual		
NIST Certified Weights	Accuracy determined by accredited weights and measurement laboratory.	1 year	As per certificate.	Replace.
NIST- Traceable Thermometer- Mercury	Accuracy determined by accredited measurement laboratory.	3 years	As per certificate.	Replace.
NIST- Traceable Thermometer- Digital	Accuracy determined by accredited measurement laboratory.	1 year	As per certificate	Replace.
Thermometer	Against NIST-traceable thermometer	Yearly at appropriate temperature range for intended use	± 1.2°C	Replace
Minimum- Maximum Thermometers	Against NIST-traceable thermometer	Yearly	± 1.5°C	Replace

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Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
InfraRed Temperature Guns	Against NIST-traceable thermometer	Daily at appropriate temperature range for intended use.	± 1.5°C	Repair/replace
	Accuracy determined by accredited measurement laboratory.	Annual		
Dial-type Thermometers	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	± 1.5°C	Replace
Refrigerator	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, check again in two hours.	0-6°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Freezer	Temperature checked using NIST-traceable thermometer	Daily. If out of range, check again in two hours.	(-10)-(-20)°C	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify supervisor.
Oven	Temperature checked using NIST-traceable thermometer.	When in use.	104 ± 1°C (drying) 180 ± 2°C (TDS)	Adjust. Replace.
Water Bath	Temperature checked using NIST-traceable thermometer.	When in use.	± 2°C	Adjust. Replace.
Volumetric Dispensing Devices (Eppendorf ® pipette, automatic dilutor or	One delivery by weight. Using DI water or solvent of use, dispense into tared vessel. Record weight with device ID number.	Each day of use	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
dispensing devices)	Calibrate using 4 replicate gravimetric measurements	Quarterly		

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Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
Glass Microliter Syringes	None	Accuracy must be initially demonstrated if syringe was not received with a certificate attesting to established accuracy.	± 1%	Not applicable.
Deionized Water	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Daily	<1.0 µmho at 25°C	Record on log. Report discrepancies to QA Manager, Operations Manager or Technical Director.

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#### **SECTION 21**

#### MEASUREMENT TRACEABILITY (NELAC 5.5.6)

#### 21.1 <u>OVERVIEW</u>

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

# 21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), (APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

The calibration report or certificate submitted to **TestAmerica Buffalo** contains, in a well designed format, a traceability statement, the conditions under which the calibrations were made in the context of any potential influence, a compliance statement with an identified metrological specification and the pertinent clauses, a clearly identified record of the quantities and functional test results before and after re-calibration, and no recommendation on the calibration interval. Opinions and interpretations of results are presented along with the basis upon which they were made and identified as such. The report may be submitted by facsimile or other electronic means as long as the requirements of the International Standard are achieved. If significant amendments are made to a calibration certificate, a supplemental certificate for the serial-number-specified piece of equipment is so identified. When a new certificate is offered, it uniquely identifies and references the one it replaces. All calibration reports are filed in the QA Office.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance

calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

# 21.3 **REFERENCE STANDARDS / MATERIALS**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA or NVLAP with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. Method specific information may also be found in the laboratory method SOPs in the "Standards and Reagents" sections. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

# 21.4 <u>DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND</u> <u>REFERENCE MATERIALS</u>

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. Refer to SOP No. CA-Q-S-001, Solvent and Acid Lot Testing and Approval.

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained by each department in bound or electronic folders. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored

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appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer laboratory SOP BF-GP-019, "Standard Traceability and Preparation" and also to the method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

**21.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory department's chemical history log and are assigned a unique identification number. Preparation of working standards or reagents prepared from the stock is documented in the laboratory Department's Standard Preparation Log. The following information is typically recorded:

- Standard ID
- Description of Standard
- Department
- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment section

Records are maintained for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID
- Special Health/Safety warnings if applicable

**21.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOPs.

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## **SECTION 22.0**

# SAMPLING

# (NELAC 5.5.7)

# 22.1 <u>OVERVIEW</u>

The laboratory provides sampling services. Sampling procedures are described in the following SOPs:

- **BF-FS-001** Chain of Custody Documentation
- BF-FS-002 Sample Packaging and Shipment Off-Site
- BF-FS-003 Groundwater Sampling Field Data Collection
- **BF-FS-004** Equipment Decontamination
- BF-FS-005 Groundwater/Surface Water Sampling
- BF-FS-006 Calibration of Field Meter
- **BF-FS-007** Low Flow Sampling Procedures
- **BF-FS-008** Surface and Subsurface Soil/Sediment Sampling

# 22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

# 22.2.1 <u>Preservatives</u>

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid Reagent ACS (Certified VOA Free) or equivalent
- Methanol Purge and Trap grade
- Nitric Acid Instra-Analyzed or equivalent
- Sodium Bisulfate ACS Grade or equivalent
- Sodium Hydroxide Instra-Analyzed or equivalent
- Sulfuric Acid Instra-Analyzed or equivalent
- Sodium Thiosulfate ACS Grade or equivalent

# 22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the chain-of-custody (COC) form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g. 14 days, 28 days), the holding time is based on calendar day measured. Holding times

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expressed in "hours" (e.g. 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time for time critical parameters ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is. These programs will be addressed on a case-by-case basis.

# 22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

The preservation and holding time criteria specified in the following tables are derived from the source documents for the methods. If method required holding times, this info is in the SOP or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

# 22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

The following information provides general guidance for homogenization and subsampling. For laboratory specific procedures refer to SOP BF-GP-005, "Sample Homogenization and Subsampling".

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# **SECTION 23**

## HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

## 23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

## 23.1.1 <u>Field Documentation</u>

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available

• The date and time that each person received or relinquished the sample(s), including their signed name.

The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The shipping documents are retained with the project files.

# 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC or in the project notes, sample management will initiate Strict Chain of Custody procedures as defined in SOP BF-GP-018, "Strict Internal Chain-of-Custody".

# 23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

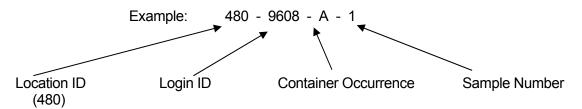
# 23.2.1 <u>Laboratory Receipt</u>

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Login – Analytical Receipt Resolution Form and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

## 23.2.1.1 Unique Sample Identification

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 4 components):



The above example states that TestAmerica Buffalo Laboratory (Location 480). Login ID is 9608 (unique to a particular client/job occurrence). The container code indicates it is the first container ("A") of Sample #1.

If the primary container goes through a prep step that creates a "new" container, then the new container is considered secondary and gets another ID. An example of this being a client sample in a 1-Liter amber bottle is sent through a Liquid/Liquid Extraction and an extraction vial is created from this step. The vial would be a SECONDARY container. The secondary ID has 5 components.

### Example: XXX - 9608 - A - 1 - A - - Secondary Container Occurrence

Example: 220-9608-A-1-A, would indicate the PRIMARY container listed above that went through a step that created the 1<sup>st</sup> occurrence of a Secondary container.

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

### 23.3 SAMPLE ACCEPTANCE POLICY

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

- **23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.
- **23.3.2** Any deviations from these checks described in Section 23.1.1.1 that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:
  - Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
  - Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP No. BF-SR-002.

# 23.4 SAMPLE STORAGE

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. Aqueous samples designated for metals analysis are stored at ambient temperature. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed at a minimum of every two weeks.

Analysts and technicians provide a request form to the cooler custodian who then retrieves the requested samples. In the absence of the cooler custodian, the analysts may personally retrieve the sample containers allocated to their analysis from the designated refrigerator. The samples are placed on carts, transported the analytical area and analyzed. Following analysis the remaining sample is returned to the refrigerator from which it originally came. All unused portions of samples are returned to the secure sample control area. All samples are kept in the refrigerators

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for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to dry room temperature, sample archive area where they are retained a minimum of 2 weeks after the final report has been issued to the client at which time disposal occurs. Special arrangements may be made to store samples for longer periods of time. Extended archival periods allow additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

# 23.5 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, samples which are known or suspected to be hazardous are segregated and a notification is issued to all laboratory personnel. All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm. All soil samples, including foreign soil samples are heat treated or incinerated in accordance with USDA permit requirements and are transported / disposed by USEPA approved facilities.

Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

# 23.6 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). For sample shipments which include water/solid volatile organic analyses (see Note), A trip blank is enclosed when required by method specifications or state or regulatory programs. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

Note: If a client does not request trip blank analysis on the COC or other paperwork, the laboratory will analyze the trip blanks that were supplied.

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# 23.7 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP: BF-WM-001, "Waste Management".) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than six weeks from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample may request to participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal and nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client). All disposal of sample containers is accomplished through incineration. A Waste Disposal Record should be completed.

# Figure 23-1.

# Example: Chain of Custody (COC)

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DISTRIBUTION: WHITE - Returned to Client with Report; CANARY - Stays with the Sample; PINK - Field Copy

### Figure 23-2.

## Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

- 1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
  - > Client name, address, phone number and fax number (if available)
  - Project name and/or number
  - > The sample identification
  - > Date, time and location of sampling
  - > The collectors name
  - > The matrix description
  - > The container description
  - > The total number of each type of container
  - > Preservatives used
  - > Analysis requested
  - Requested turnaround time (TAT)
  - > Any special instructions
  - > Purchase Order number or billing information (e.g. quote number) if available
  - The date and time that each person received or relinquished the sample(s), including their signed name.
  - > The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
  - Information must be legible
- 2) Samples must be properly labeled.
  - Use durable labels (labels provided by TestAmerica are preferred)
  - Include a unique identification number
  - Include sampling date and time & sampler ID
  - Include preservative used.
  - Use indelible ink
  - > Information must be legible
- 3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.
- 4) Samples must be preserved according to the requirements of the requested analytical method. See lab Sampling Guide.

**Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
- For Volatile Organic analyses in drinking water (Methods 502.2 or 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
  - > 1. Test for residual chlorine in the field prior to sampling.
    - > If no chlorine is present, the samples are to be preserved using HCl as usual.
    - If chlorine is present, add either ascorbic acid or sodium thiosulfate prior to adding HCI.
  - 2. Use VOA vials pre-preserved with sodium thiosulfate or ascorbic acid and add HCI after filling the VOA vial with the sample.
- FOR WATER SAMPLES TESTED FOR CYANIDE for NPDES samples by Standard Methods or EPA 335
  - In the Field: Samples are to be tested for Sulfide using lead acetate paper prior to the addition of Sodium Hydroxide (NaOH). If sulfide is present, the sample must be treated with Cadmium Chloride and filtered prior to the addition of NaOH.
    - If the sulfide test and treatment is not performed in the field, the lab will test the samples for sulfide using lead acetate paper at the time of receipt and if sulfide is present in the sample, the client will be notified and given the option of retaking the sample and treating in the field per the method requirements or the laboratory can analyze the samples as delivered and qualify the results in the final report.
  - It is the responsibility of the client to notify the laboratory if thiosulfate, sulfite, or thiocyanate are known or suspected to be present in the sample. This notification may be on the chain of custody. The samples may need to be subcontracted to a laboratory that performs a UV digestion. If the lab does not perform the UV digestion on samples that contain these compounds, the results must be qualified in the final report.
  - The laboratory must test the sample for oxidizing agents (e.g. Chlorine) prior to analysis and treat according to the methods prior to distillation. (ascorbic acid or sodium arsenite are the preferred choice).
- 5) Sample Holding Times
  - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (2 working days) remaining on the holding time to ensure analysis.</p>
  - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis.
- 6) All samples submitted for Volatile Organic analyses must have a Trip Blank submitted at the same time. TestAmerica will supply this blank with the bottle order.

- 7) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.
- 8) Recommendations for packing samples for shipment.
  - > Pack samples in Ice rather than "Blue" ice packs.
  - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
  - Water samples would be best if wrapped with bubble-wrap or paper (newspaper, or paper towels work) and then placed in plastic zip-lock bags.
  - > Fill extra cooler space with bubble wrap.

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Figure 23-3. Example: Cooler Receipt Form TestAmerica Buffalo	Doc. Login Front Rev 8 10/15/2010
WORK ORDER RT	
CLIENT PROJECT	Γ
Pre-Log Strict Inte	ernal COC: YES / NO
TATBD/CD # OF SAMPLES	5TRIP BLANK Y/N #
SHIPPED BY	ATTACH SHIPPING TAGS(back)
RECEIVED DATE / TIME:	/:::
COOLER TEMP °C (<6 °C)	OK NO SEE BACK
Cooler Custody Seal intact? YES/NO NONE	SEAL #
If NO to cooler temp or seal, PM notified? YES_	(PM Name)
Sample received outside hold time	
Condition/Issues YES/NO	ARRF
Resolved at login	
PRESERVATION CHECKED YES NO	_ NA Initials
RESIDUAL CHLORINE CHECK: VES, OK V	ES, Qualified $\square$ NO, no volume $\square$ NA
RADIATION CHECK <0.02 mR/hr: YES/ NO	
ARE SAMPLE DATES AND TIMES CORRECT?	? Initials
WERE ALL THE APPROPRIATE TESTS ASSIG	GNED? Initials
Temp Cert Loss:	

### SECTION 24.0

# ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

## 24.1 <u>OVERVIEW</u>

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

# 24.2 <u>CONTROLS</u>

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

# 24.3 NEGATIVE CONTROLS

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.
	The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1.

Table 24-1.

Control Turne	Detaile
Control Type	Details
Trip Blank <sup>1</sup>	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks <sup>1</sup>	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks <sup>1</sup>	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

# 24.4 **POSITIVE CONTROLS**

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 <u>Method Performance Control - Laboratory Control Sample (LCS)</u>

- **24.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.
- **24.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the

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associated samples. The LCS is spiked with verified known amounts of analytes or is made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard may be reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

- **24.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- **24.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- **24.4.1.5** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.
  - **24.4.1.5.1** For methods that have 1-10 target analytes, spike all components.
  - **24.4.1.5.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
  - **24.4.1.5.3** For methods with more than 20 target analytes, spike at least 16 components.
  - **24.4.1.5.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
  - **24.4.1.5.5** Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

# 24.5 SAMPLE MATRIX CONTROLS

#### Table 24-5. Sample Matrix Control

Control Type		Details
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency <sup>1</sup>	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency <sup>1</sup>	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

# 24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

**24.6.1** As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**24.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**24.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm$  3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

**24.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).

**24.6.3.2** In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.

- **24.6.3.3** The lowest acceptable recovery limit will be 10% (the analyte must be detectable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable.
- **24.6.3.4** The maximum acceptable recovery limit will be 150%.

**24.6.3.5** The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.

**24.6.3.6** If either the high or low end of the control limit changes by  $\leq 5\%$  from previous, the data points are inspected and, using professional judgment, the limits may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits.

**24.6.4.1** The control limits are maintained in the laboratory LIMs system. The limits for each analyte/method/matrix combination are assigned effective and expiration dates. The QA department is able to query the LIMs system and print an active list of control limits based on this database. The most current laboratory limits (based on the effective/expiration dates) are reflected on the laboratory worksheets and final reports unless superseded by project specific limits.

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**24.6.5** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 13) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- **24.6.5.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.
- **24.6.5.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

**24.6.6** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.7** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

# 24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

**24.7.1** The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples.

**24.7.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

**24.7.3** Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

**24.7.4** Selection of appropriate reagents and standards is included in Section 9 and 22.

**24.7.5** A discussion on selectivity of the test is included in Section 5.

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- **24.7.6** Constant and consistent test conditions are discussed in Section 19.
- **24.7.7** The laboratories sample acceptance policy is included in Section 23.

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#### SECTION 25.0

#### REPORTING RESULTS (NELAC 5.5.10)

#### 25.1 <u>OVERVIEW</u>

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. A variety of report formats are available to meet specific needs. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

### 25.2 <u>TEST REPORTS</u>

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed on laboratory letterhead, reviewed, and signed by the appropriate project manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report) with a "sample results" column header.

**25.2.2** Each report cover page is printed on company letterhead which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as # / ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC).

• Any COCs involved with Subcontracting are included.

- In most cases, the applicable COC is paginated and is an integral part of the report.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).

**25.2.5** The name and address of client and a project name/number, if applicable.

**25.2.6** Client project manager or other contact

**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

- **25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).
- **25.2.11** Practical quantitation limits or client reporting limit.
- **25.2.12** Method detection limits (if requested)
- **25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).
- **25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested).

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda). Sample temperatures are recorded in the report case narrative and on the COC. Deviations from normal conditions (e.g., preservation, breakage) are recorded in the report case narrative.

**25.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

**25.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

**25.2.20** When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

**25.2.21** The laboratory includes a cover letter.

**25.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.23** When Soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

**25.2.24** Appropriate laboratory certification number for the state of origin of the sample if applicable.

**25.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g, partial report). A complete report must be sent once all of the work has been completed.

**25.2.26** Any non-TestAmerica subcontracted analysis results are provided as an addendum to the report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

# 25.3 <u>REPORTING LEVEL OR REPORT TYPE</u>

**TestAmerica Buffalo** offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 26.7.

## 25.3.1 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. *TestAmerica Buffalo* offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), Excel, Dbase, GISKEY, and Text Files.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

## 25.4 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report

**25.4.1** Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

**25.4.2** Where quality system requirements are not met, a statement of compliance/noncompliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

**25.4.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**25.4.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

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**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

# 25.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in Section 8.

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

# 25.6 <u>CLIENT CONFIDENTIALITY</u>

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information <u>known</u> to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**25.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. It is our policy that facsimiles are intended for and should be used for business purposes only. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this

communication is strictly prohibited. If you have received this communication in error, please notify the sender.

# 25.7 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

# 25.8 AMENDMENTS TO TEST REPORTS

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "R". The revised report will have the word "revised" appended to the cover letter.

When the report is re-issued, a notation of "revised" is placed on the cover/signature page of the report. A brief explanation of reason for the re-issue is included in the report case narrative.

# 25.9 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

# 25.9.1 Policy on Data Omissions or Reporting Limit Increases

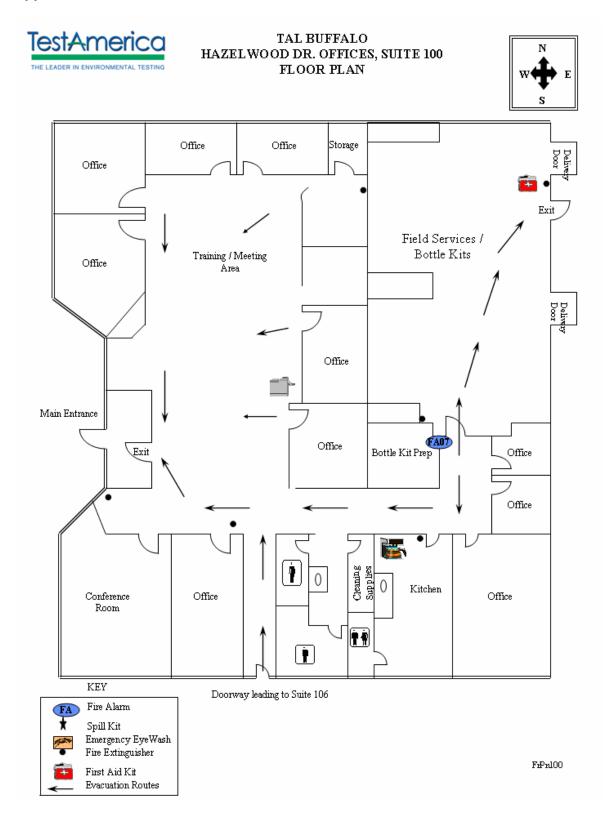
Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely <u>no possible</u> impact on the interpretation of the analytical results and there is <u>no possibility</u> of the change being interpreted as misrepresentation by anyone inside or outside of our company.

# 25.9.2 <u>Multiple Reports</u>

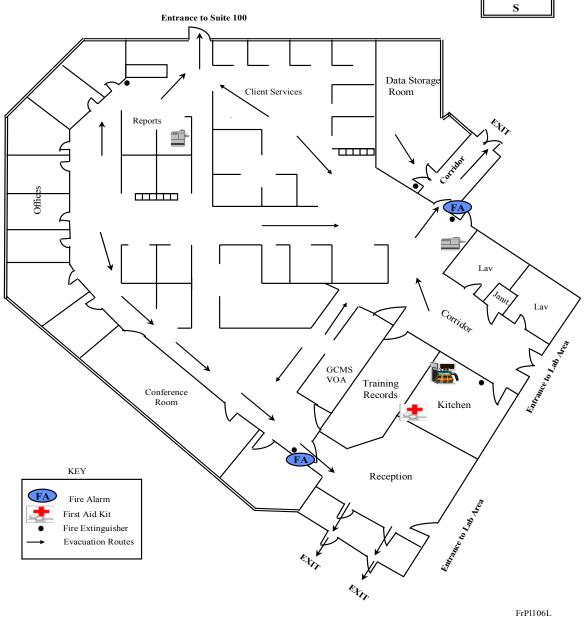
TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

# Appendix 1.



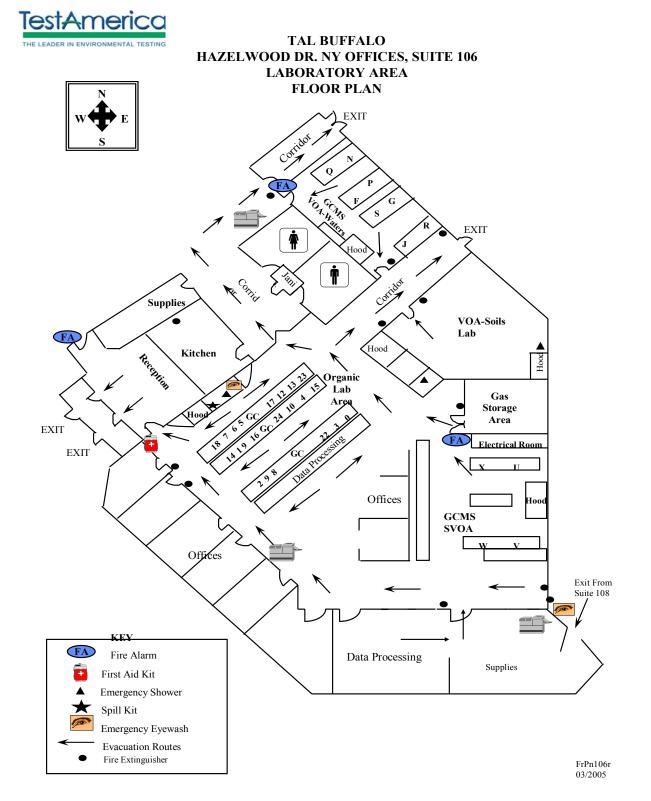


#### TAL BUFFALO HAZELWOOD DR. OFFICES, SUITE 106 CLIENT SERVICES/REPORT PREP FLOOR PLAN

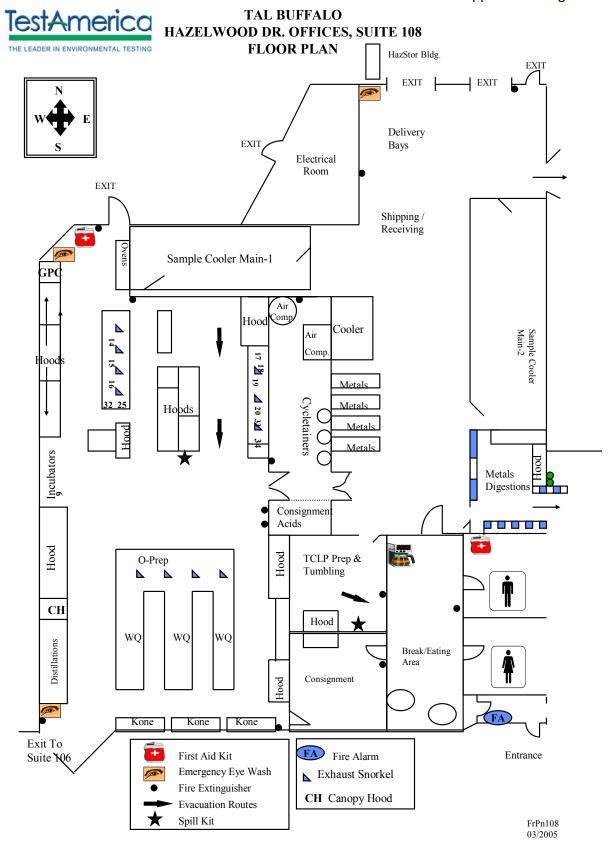


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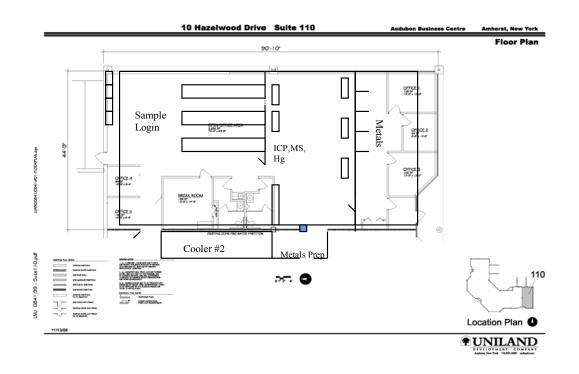
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### Appendix 2. Glossary/Acronyms

#### Glossary:

#### Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

#### Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

#### Accrediting Authority:

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

#### Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

#### Analyst:

The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

#### Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

#### Blank:

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

Blind Sample:

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

#### Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

#### Calibration Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Method:

A defined technical procedure for performing a calibration. (NELAC)

Calibration Standard:

A substance or reference material used to calibrate an instrument (QAMS)

Certified Reference Material (CRM):

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

Chain of Custody:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

Clean Air Act:

The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):

The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

### Compromised Samples:

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

### Confidential Business Information (CBI):

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its

representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

#### Confirmation:

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation Alternate wavelength Derivitization Mass spectral interpretation Alternative detectors or Additional Cleanup procedures

(NELAC)

#### Conformance:

An affirmative indication or judgment that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

#### Corrective Action:

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

#### Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

#### Data Reduction:

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

#### Deficiency:

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

### Detection Limit:

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

#### Document Control:

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

#### Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

#### Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

#### External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

Field Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

#### Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

#### Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intralaboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

Least Squares Regression (1<sup>st</sup> Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for Inorganics.

#### Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

Matrix:

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

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Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% Settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% Settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

#### Method Detection Limit:

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

#### Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

#### Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Performance Based Measurement System (PBMS):

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

#### Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

#### Proficiency Testing:

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

#### Proficiency Testing Program:

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

#### Proficiency Test Sample (PT):

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

#### Quality Assurance:

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

#### Quality Assurance [Project] Plan (QAPP):

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

#### Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

#### Quality Control Sample:

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

#### Quality Manual:

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

#### Quality System:

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

#### Quantitation Limits:

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

#### Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

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Reagent Blank (method reagent blank):

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

#### Reference Material:

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

#### Reference Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

#### Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

#### Report Limit (RL):

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

#### Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

#### Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

#### Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

Second Order Polynomial Curve (Quadratic): The  $2^{nd}$  order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The  $2^{nd}$  order regression will generate a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.99.

#### Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

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#### Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

#### Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

#### Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

#### Standard Operating Procedures (SOPs):

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

#### Standardized Reference Material (SRM):

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

#### Surrogate:

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

#### Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

### Acronyms:

BS – Blank Spike BSD – Blank Spike Duplicate CAR – Corrective Action Report CCV - Continuing Calibration Verification CF – Calibration Factor CFR – Code of Federal Regulations COC – Chain of Custody CRS – Change Request Form DOC – Demonstration of Capability DQO - Data Quality Objectives DU – Duplicate **DUP** - Duplicate EHS - Environment, Health and Safety EPA – Environmental Protection Agency GC - Gas Chromatography GC/MS - Gas Chromatography/Mass Spectrometry HPLC - High Performance Liquid Chromatography ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy ICV – Initial Calibration Verification IDL – Instrument Detection Limit IH – Industrial Hygiene IS – Internal Standard LCS - Laboratory Control Sample LCSD – Laboratory Control Sample Duplicate LIMS – Laboratory Information Management System MDL – Method Detection Limit MS - Matrix Spike MSD – Matrix Spike Duplicate MSDS - Material Safety Data Sheet NELAC - National Environmental Laboratory Accreditation Conference NELAP - National Environmental Laboratory Accreditation Program PT – Performance Testing QAM – Quality Assurance Manual QA/QC – Quality Assurance / Quality Control QAPP – Quality Assurance Project Plan **RF** – Response Factor **RPD** – Relative Percent Difference RSD - Relative Standard Deviation SD – Standard Deviation SOP: Standard Operating Procedure TAT - Turn-Around-Time VOA – Volatiles VOC – Volatile Organic Compound

#### Appendix 3.

#### Laboratory Certifications, Accreditations, Validations

**TestAmerica Buffalo** maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

OTATE	Dream and	Cert # / Lab ID
STATE	Program	
Arkansas	CWA, RCRA, SOIL	88-0686
California*	NELAP CWA, RCRA	01169CA
Connecticut	SDWA, CWA, RCRA, SOIL	PH-0568
Florida*	NELAP CWA, RCRA	E87672
Georgia*	SDWA,NELAP CWA, RCRA	956
Illinois*	NELAP SDWA, CWA, RCRA	200003
Iowa	SW/CS	374
Kansas*	NELAP SDWA, CWA, RCRA	E-10187
Kentucky	SDWA	90029
Kentucky UST	UST	30
Louisiana*	NELAP CWA, RCRA	2031
Maine	SDWA, CWA	NY0044
Maryland	SDWA	294
Massachusetts	SDWA, CWA	M-NY044
Michigan	SDWA	9937
Minnesota	SDWA,CWA, RCRA	036-999-337
New Hampshire*	NELAP SDWA, CWA	233701
New Jersey*	NELAP,SDWA, CWA, RCRA,	NY455
New York*	NELAP, AIR, SDWA, CWA, RCRA	10026
North Dakota	CWA, RCRA	R-176
Oklahoma	CWA, RCRA	9421
Oregon*	CWA,RCRA	NY200003
Pennsylvania*	NELAP CWA,RCRA	68-00281
Tennessee	SDWA	02970
Texas*	NELAP CWA, RCRA	T104704412-08-TX
USDA	FOREIGN SOIL PERMIT	S-41579
Virginia	SDWA	278
Washington*	NELAP CWA,RCRA	C1677
Wisconsin	CWA, RCRA	998310390
West Virginia	CWA,RCRA	252

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, and in the QA Department.

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THE LEADER IN ENVIRONMENTAL TESTING

# **Cover Page:**

# **Quality Assurance Manual**

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# **Title Page:**

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12/3/28

Date

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# **REFERENCED CORPORATE SOPs AND POLICIES**

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

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# **REFERENCED LABORATORY SOPs**

SOP Reference	Title
CT-QAS-3	SOP for Document Control (Sec. 3.4.1)
CT-MKS-7	Complaint Resolution (Sec .10.1)
QAF00810 QAW01200/1300/1400 QAW1700 QAW01500 QAW01600	SOP Listing (Sec. 13.2) PT Tracking Equipment Table Master Certification Spreadsheet Personnel & Qualifications
CT-RPS-7	LIMS Final Reporting (Sec. 14.1.4)
CT-QAS-16	SOP for Employee Training (Sec. 17.3)
CT-QAS-8	Generating SOPs (Sec. 19.2)
CT-QAS-27	Demonstration of Capability (Sec. 19.4.2)
CT-QAS-17	Conducting MDL Studies (Sec. 19.7)
CT-SMS-11	SOP for Compositing, Homogenization and Subsampling Environmental Samples (Sec. 22.5)
CT-SMS-4	Sample Processing at Sample Arrival (Sec. 23.2.1.3)

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#### **SECTION 3**

#### INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

TestAmerica Connecticut's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with the 2003 National Environmental Laboratory Accreditation Conference (NELAC) standards and ISO/IEC Guide 17025 (1999). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan(CQMP) and the various accreditation and certification programs listed in Appendix 3. The relevant NELAC section is included in the heading of each QAM section.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/R-95/131, *Methods for the Determination of Organic Compounds in Drinking Water,* Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- EPA SW-846, *Test Methods for the Evaluation of Solid Waste, 3<sup>rd</sup> Edition,* September 1986; Update I, July 1992; Update II, September 1994; and Update III, December 1996.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- USEPA Contract Laboratory Program. *Statement of Work for Inorganics Analysis. Multi-Media, Multi-Concentration*. Document ILM04.0.
- USEPA Contract Laboratory Program. *Statement of Work for Organics Analysis. Multi-Media, Multi-Concentration.* Document Number OLMO3.1, August 1994, OLM04.2.
- APHA, *Standard Methods for the Examination of Water and Wastewater,* 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and 21<sup>st</sup> Edition.
- Nuclear Regulatory Commission (NRC) quality assurance requirements.

#### 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

# 3.3 SCOPE / FIELDS OF TESTING

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge and soils. The Quality Assurance Program contains specific procedures and methods to test samples of differing matrices for chemical, physical parameters. The Program also contains guidelines on maintaining documentation of analytical process, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all requests to provide analyses are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4 of the QAM. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director/Manager and the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director/Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

# 3.4 MANAGEMENT OF THE MANUAL

# 3.4.1 <u>Review Process</u>

This manual is reviewed annually by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control & Updating procedures (refer to SOP No. CT-QAS-3).

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#### **SECTION 4**

#### ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 <u>OVERVIEW</u>

TestAmerica Connecticut is a local operating unit of TestAmerica Laboratories, Inc.. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. TestAmerica Connecticut has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Operating Officer, Corporate Quality Assurance, etc.). The TestAmerica Connecticut laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Connecticut is presented in Figure 4-1.

### 4.2 ROLES AND RESPONSIBILITIES

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### 4.2.1 Quality Assurance Program

The responsibility for quality lies with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the CQMP. This manual is specific to the operations of TestAmerica's Connecticut laboratory.

#### 4.2.2 <u>General Manager (GM)</u>

Each GM reports directly to a COO. Each GM has full responsibility for the overall administrative and operational management of their respective laboratories. The GM's responsibilities include allocation of personnel and resources, long-term planning, setting goals, and achieving the financial, business, and quality objectives of TestAmerica. The GM ensures timely compliance with corporate management directives, policies, and management systems reviews. The GM is also responsible for restricting any laboratory from performing analyses that cannot be consistently and successfully performed to meet the standards set forth in this manual.

# 4.2.3 <u>Laboratory Director</u>

TestAmerica Connecticut's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program.

Specific responsibilities include, but are not limited to:

- Provides one or more technical directors for the appropriate fields of testing. The name(s) of the Technical Director will be included in the national database. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, the Laboratory Director must designate another full time staff member meeting the qualifications of the Technical Director to temporarily perform this function. If the absence exceeds 65 consecutive calendar days, the primary accrediting authority must be notified in writing.
- Ensures that all analysts and supervisors have the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Captains the management team, consisting of the QA Manager, CSM and the Technical Director(s), as direct reports.

# 4.2.4 Quality Assurance (QA) Manager

The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (i.e., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Having functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintaining and updating the QAM.
- Monitoring and evaluating laboratory certifications; scheduling proficiency testing samples.
- Monitoring and communicating regulatory changes that may affect the laboratory to management.

- Training and advising the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Having a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).
- Arranging for or conducting internal audits on quality systems and the technical operation.
- The laboratory QA Manager will maintain records of all ethics-related training, including the type and proof of attendance.
- Maintain, improve, and evaluate the corrective action database and the corrective and preventive action systems.
- Notifying laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs are temporarily suspended following the procedures outlined in Section 13.
- Monitoring standards of performance in quality control and quality assurance.
- Coordinating of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Review a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Review of external audit reports and data validation requests.
- Follow-up with audits to ensure client QAPP requirements are met.
- Establishment of reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Development of suggestions and recommendations to improve quality systems.
- Research of current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.

# 4.2.5 <u>Supervisors</u>

Supervisors (Technical Directors) report to the Laboratory Director. Each one is responsible to:

- Ensure that analysts in their department adhere to applicable SOPs and the QA Manual. They perform frequent SOP and QA Manual review to determine if analysts are in compliance and if new, modified, and optimized measures are feasible and should be added to these documents.
- With regard to analysts, participates in the selection, training (as documented in Section 8.1), development of performance objectives and standards of performance, appraisal (measurement of objectives), scheduling, counseling, discipline, and motivation of analysts and documents these activities in accordance with systems developed by the QA and Personnel Departments. They evaluate staffing sufficiency and overtime needs. Training consists of familiarization with SOP, QC, Safety, and computer systems.

- Encourage the development of analysts to become cross-trained in various methods and/or operate multiple instruments efficiently while performing maintenance and documentation, self-supervise, and function as a department team.
- Provide guidance to analysts in resolving problems encountered daily during sample prep/analysis in conjunction with the Technical Director, Operations Manager, and/or QA Manager. Each is responsible for 100% of the data review and documentation, non-conformance and CPAR issues, the timely and accurate completion of performance evaluation samples and MDLs, for his department.
- Ensure all logbooks are maintained, current, and properly labeled or archived.
- Report all non-conformance conditions to the QA Manager and Laboratory Director.
- Ensure that preventive maintenance is performed on instrumentation as detailed in the QA Manual or SOPs. He is responsible for developing and implementing a system for preventive maintenance, troubleshooting, and repairing or arranging for repair of instruments.
- Maintain adequate and valid inventory of reagents, standards, spare parts, and other relevant resources required to perform daily analysis.
- Achieve optimum turnaround time on analyses and compliance with holding times.
- Conduct efficiency and cost control evaluations on an ongoing basis to determine optimization of labor, supplies, overtime, first-run yield, capacity (designed vs. demonstrated), second- and third-generation production techniques/instruments, and long-term needs for budgetary planning.
- Develop, implement, and enhance calibration programs.
- Provide written responses to external and internal audit issues.

#### 4.2.6 <u>Laboratory Analysts</u>

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the group leader or supervisor. The responsibilities of the analysts are listed below:

- Perform analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Document standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on worklists, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Report all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their supervisor, the Technical Director, and/or the QA Manager or member of QA staff.
- Perform 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggest method improvements to their supervisor, the Technical Director, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.

 Work cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.

# 4.2.7 Environmental Health and Safety Officer

The Safety Officer reports to the Laboratory Director and ensures that systems are maintained for the safe operation of the laboratory. The Safety Officer is responsible to:

- Conduct ongoing, necessary safety training and conduct new employee safety orientation.
- Assist in developing and maintaining the Chemical Hygiene/Safety Manual.
- Administer dispersal of all Material Safety Data Sheet (MSDS) information.
- Perform regular chemical hygiene and housekeeping instruction.
- Give instruction on proper labeling and practice.
- Serve as chairman of the laboratory safety committee.
- Provide and train personnel on protective equipment.
- Oversee the inspection and maintenance of general safety equipment fire extinguishers, safety showers, eyewash fountains, etc. and ensure prompt repairs as needed.
- Supervise and schedule fire drills and emergency evacuation drills.
- Determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- When determined necessary, conduct exposure monitoring assessments.
- Determine when a complaint of possible over-exposure is "reasonable" and should be referred for medical consultation.
- Assist in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica's medical consultants.

# 4.2.8 Hazardous Waste Coordinator

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Staying current with the hazardous waste regulations.
- Continuing training on hazardous waste issues.
- Reviewing and updating annually the Hazardous Waste Contingency Plan in the Environmental Health & Safety Manual.
- Auditing the staff with regard to compliance with the Hazardous Waste Contingency Plan.
- Contacting the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.

#### 4.2.9 <u>Client Service Manager</u>

The Client Service Manager reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.
- Responsible to ensure that clients receive the proper sampling supplies.
- Accountable for response to client inquiries concerning sample status.
- Responsible for assistance to clients regarding the resolution of problems concerning COC.
- Ensuring that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifying the supervisors of incoming projects and sample delivery schedules.
- Accountable to clients for communicating sample progress in daily status meeting with agreed-upon due dates.
- Responsible for discussing with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Responsible for staff familiarization with specific quotes, sample log-in review, and final report completeness.
- Monitor the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Inform clients of data package-related problems and resolve service issues.
- Coordinate requests for sample containers and other services (data packages).

# 4.2.10 Project Manager

The Project Manager reports directly to the Division Manager and serves as liaison between the laboratory and its clients. The Project Manager's responsibilities include:

• Ensure client specifications are met by communicating project and quality assurance requirements to the laboratory. Ensure client specific reporting and quality control requirements are met.

- Notify laboratory personnel of incoming projects and sample delivery schedules.
- Monitor the status of all projects in-house to ensure timely delivery of reports.

• Inform clients of project-related problems, resolving service issues and coordinating technical issues with the laboratory staff.

- Coordinate client requests for sample containers and other services.
- Schedule sample pick-ups from client offices or project sites and notifying the laboratory staff of incoming samples.
- Coordinate subcontract work.
- Assist clients in procuring the proper sampling supplies.
- Respond to client inquiries concerning sample status.
- Assist clients with resolution of problems concerning Chains-of-Custody.

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- Prepare laboratory quotes and project bids.
- Review sample log-in sheets, when there is a question regarding a Chain of Custody issue.

# 4.2.11 Sample Custodian

The Sample Custodian reports to the Project Management Department. The responsibilities of the Sample Manager are outlined below:

- Direct the logging of incoming samples into the LIMS.
- Insure timely and correct shipment of sample containers to clients. Maintain accurate records of sample container shipments.
- Perform sample collection and sample pick-up
- Ensures sample containers are prepared for sampling

• Performs field tests and measurements and operates and maintains equipment used for those purposes.

# 4.3 <u>DEPUTIES</u>

The following table defines who assumes the responsibilities of key personnel in their absence:

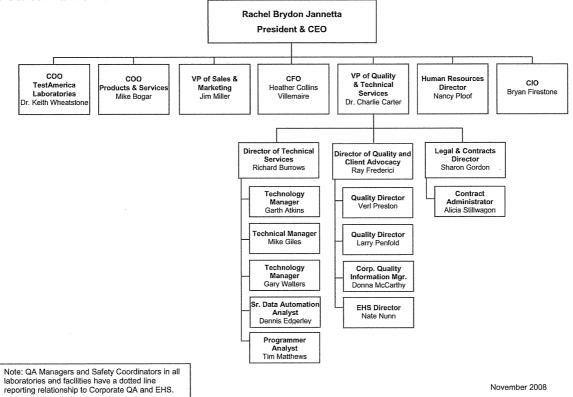
Key Personnel	Deputy	Comment
Laboratory Director Larry Decker	Paul Hobart	
QA Manager Dawn May	Patty Mercure	
Organic Technical Director Kim Maturo	Magdalena Szymczuk	
Metals Technical Director Nestor Petronchak	Joseph Voytek	
Wet Chemistry Technical Director Doreen Nemeth	David Madumadu	
EHS Coordinator Daniel Helfrich	Joseph Voytek	

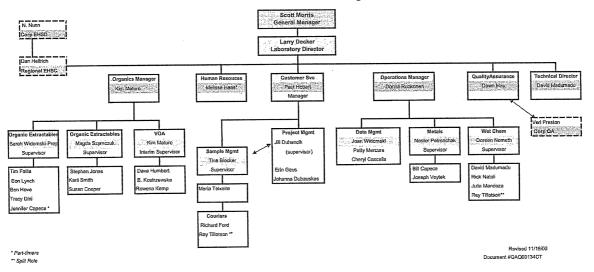
#### Figure 4-1.

**Corporate and Laboratory Organization Charts** 









#### **TestAmerica Connecticut Organization**

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#### **SECTION 5**

## QUALITY SYSTEM (NELAC 5.4.2)

### 5.1 QUALITY POLICY STATEMENT

It is TestAmerica's Policy to:

- Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- Provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

# 5.2 ETHICS AND DATA INTEGRITY

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CA-L-P-001) and Employee Ethics Statements.
- An Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CA-L-S-001.)
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CA-L-S-001).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client predefined Data Quality Objectives (DQOs).
- Present services in a confidential, honest and forthright manner.

- Provide employees with guidelines and an understanding of the Ethical and Quality Standards of our Industry.
- Operate our facilities in a manner that protects the environment and the health and safety of employees and the public.
- Obey all pertinent federal, state and local laws and regulations and encourage other members of our industry to do the same.
- Educate clients as to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

# 5.3 QUALITY SYSTEM DOCUMENTATION

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual Each laboratory has a lab specific quality assurance manual.
- <u>Corporate SOPs and Policies</u> Corporate SOPs and Policies are developed for use by all relevant laboratories. They are incorporated into the laboratory's normal SOP distribution, training and tracking system. Corporate SOPs may be general or technical.
- <u>Work Instructions</u> A subset of procedural steps, tasks or forms associated with an operation of a management system (e.g., checklists, preformatted bench sheets, forms).
- Laboratory SOPs General and Technical
- <u>Corporate Quality Policy Memorandums</u>

# 5.3.1 Order of Precedence

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Policy Memorandum
- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory's has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's (QAM) shall take precedence over the CQMP in those cases.

# 5.4 QA/QC OBJECTIVES FOR THE MEASUREMENT OF DATA

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term *"analytical quality control"*. QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS).

# 5.4.1 <u>Precision</u>

The laboratory objective for precision is to meet the performance for precision demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Precision is defined as the degree of reproducibility of measurements under a given set of analytical conditions (exclusive of field sampling variability). Precision is documented on the basis of replicate analysis, usually duplicate or matrix spike (MS) duplicate samples.

# 5.4.2 <u>Accuracy</u>

The laboratory objective for accuracy is to meet the performance for accuracy demonstrated for the methods on similar samples and to meet data quality objectives of the EPA and/or other regulatory programs. Accuracy is defined as the degree of bias in a measurement system. Accuracy may be documented through the use of laboratory control samples (LCS) and/or MS. A statement of accuracy is expressed as an interval of acceptance recovery about the mean recovery.

# 5.4.3 <u>Representativeness</u>

The laboratory objective for representativeness is to provide data which is representative of the sampled medium. Representativeness is defined as the degree to which data represent a characteristic of a population or set of samples and is a measurement of both analytical and field sampling precision. The representativeness of the analytical data is a function of the procedures used in procuring and processing the samples. The representativeness can be

documented by the relative percent difference between separately procured, but otherwise identical samples or sample aliquots.

The representativeness of the data from the sampling sites depends on both the sampling procedures and the analytical procedures. The laboratory may provide guidance to the client regarding proper sampling and handling methods in order to assure the integrity of the samples.

# 5.4.4 <u>Comparability</u>

The comparability objective is to provide analytical data for which the accuracy, precision, representativeness and reporting limit statistics are similar to these quality indicators generated by other laboratories for similar samples, and data generated by the laboratory over time.

The comparability objective is documented by inter-laboratory studies carried out by regulatory agencies or carried out for specific projects or contracts, by comparison of periodically generated statements of accuracy, precision and reporting limits with those of other laboratories.

### 5.4.5 <u>Completeness</u>

The completeness objective for data is 90% (or as specified by a particular project), expressed as the ratio of the valid data to the total data over the course of the project. Data will be considered valid if they are adequate for their intended use. Data usability will be defined in a QAPP, project scope or regulatory requirement. Data validation is the process for reviewing data to determine its usability and completeness. If the completeness objective is not met, actions will be taken internally and with the data user to improve performance. This may take the form of an audit to evaluate the methodology and procedures as possible sources for the difficulty or may result in a recommendation to use a different method.

# 5.4.6 <u>Selectivity</u>

Selectivity is defined as: The capability of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. Target analytes are separated from non-target constituents and subsequently identified/detected through one or more of the following, depending on the analytical method: extractions (separation), digestions (separation), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

## 5.4.7 <u>Sensitivity</u>

Sensitivity refers to the amount of analyte necessary to produce a detector response that can be reliably detected (Method Detection Limit) or quantified (Reporting Limit).

#### 5.5 CRITERIA FOR QUALITY INDICATORS

The laboratory maintains a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for performed analyses. This summary includes

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an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from US EPA methods when they are required. Where US EPA method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits is contained in Section 24.

## 5.6 STATISTICAL QUALITY CONTROL

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as the Ohio Voluntary Action Plan (VAP)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Supervisor and QA Manager) and entered into the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory. All control limits are stored within a LIMS Method limit group. These are set up under the control of the QA department. Any time a limit is updted, a historical record with activiation and expiration date is generated for the limit type. Archived limits can be exported to excel at any time by utilizing the "Historical" button in the Method limit group. If a method defines the QC limits, the method limits are used.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

Surrogate recoveries are determined for a specific time period as defined above. The resulting ranges are entered in LIMS.

Current QC limits are entered and maintained in the LIMS analyte database. As sample results and the related QC are entered into LIMS, the sample QC values are compared with the limits in LIMS to determine if they are within the acceptable range. The analyst then evaluates if the sample needs to be rerun or re-extracted/rerun or if a comment should be added to the report explaining the reason for the QC outlier.

# 5.6.1 <u>QC Charts</u>

Control charts are used to establish method performance of a given analysis and to monitor trends of QC results graphically over time. Once a data base of the laboratory results for a method/matrix/QC analyte combination is established, the acceptability of a given analysis of that QC parameter (and of the analytical batch to which it belongs) can be evaluated in light of the laboratory's normal performance. This is intended to help identify problems before they might affect data. Often, patterns of response that are not at all evident in sets of numbers are very distinct when the same values are viewed as a chronological graph.

#### Establishment of Limits

The purpose of using statistical control limits is to define, for each analyte in a given method/matrix/QC type combination, a range of expected values. This range encompasses the random variation that occurs normally in the laboratory and allows one to evaluate control samples

in that context, rather than according to an arbitrary or external set of values. Limits for accuracy and precision are defined below:

#### <u>Accuracy</u>

As recoveries of a QC analyte in a given matrix are tabulated over time, a mean value for recovery is established, as is the standard deviation (s) of those recoveries. If the analysis is in statistical control (e.g., if the set of QC recoveries over time show random variation about the mean) approximately 99.7% of all recoveries for that QC will fall within three standard deviations (3s) of the mean. Thus, assuming that the mean itself is an acceptable level of recovery, the values corresponding to 3s above and 3s below the mean are defined as the Control Limits. Any single recovery outside these values is assumed to have resulted from some circumstance other than normal variation and shall be investigated.

Roughly 95% of points should fall within 2s of the mean. The values +2s and -2s are the Warning Limits. Any normal result has approximately a 1/20 chance of being between 2s and 3s from the mean, so a result in this region doesn't necessarily warrant corrective action, but attention should be paid to such points.

## **Precision**

Precision is used to indicate matrix variability so that appropriate decisions can be made by the client when repeated analyses vary significantly. The coefficient of variation, expressed as a percentage (e.g., the %RSD) for the data set used to calculate accuracy control limits defines the control limit for precision. Duplicate analyses of the QC samples, such as duplicates or MS/MSD, should have an RPD less than or equal to this established precision control limit to be considered free of matrix interferences.

# 5.7 QUALITY SYSTEM METRICS

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

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### **SECTION 6**

## DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 <u>OVERVIEW</u>

The QA Department is responsible for the control of documents used in the laboratory to ensure that approved, up-to-date documents are in circulation and out-of-date (obsolete) documents are archived or destroyed. The following documents, at a minimum, must be controlled:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP No. CT-QAS-3.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

## 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a document control system for each document include a unique document title and number, the number of pages of the item, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department. In order to develop a new document, a manager submits an electronic draft to the QA Department for suggestions and approval before use. Upon approval, QA personnel add the identifying version information to the document and retains the official document on file. The official document is provided to all applicable operational units (may include electronic access). Controlled documents are

identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every two years and revised as appropriate. Changes to documents occur when a procedural change warrants.

# 6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY

For changes to the QA Manual, refer to SOP on Document Control, SOP No. CT-QAS-3. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP No. CT-QAS-008, Standard Operating Procedure for Generating SOPs. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized by department in the QA office. These are tracked by excel spreadsheet. Electronic versions are kept on the laboratory server; hard copies are kept in QA files. The procedure for the care of these documents is in SOP CT-QAS-3, SOP for Document Control.

# 6.4 OBSOLETE DOCUMENTS

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. In general, obsolete documents are collected from employees according to distribution lists and are marked obsolete on the cover or destroyed. At least one copy of the obsolete document is archived according to the SOP on Document Control SOP No. CT-QAS-3.

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#### **SECTION 7**

## SERVICE TO THE CLIENT (NELAC 5.4.7)

## 7.1 <u>OVERVIEW</u>

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these regulatory and client requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the lab's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record.

The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## 7.2 REVIEW SEQUENCE AND KEY PERSONNEL

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP No. CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below) :

- Legal & Contracts Director
- General Manager
- The Laboratory Client Service Manager
- The Laboratory Operations Manager
- Laboratory and/or Corporate Technical Directors
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director/Manager reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The PM assigned to the project maintains a copy for the lab.

# 7.3 DOCUMENTATION

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. All relevant information is stored on a designated corporate server in the contracts\Connecticut directory.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel and the Regional Account Manager. A copy of the contract and formal quote will be filed with the laboratory PM and the Lab Director/Manager.

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log in a note book of pertinent conversations with the client. If need be, a follow up email is sent.

# 7.3.1 Project-Specific Quality Planning

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA department involvement may be needed to assist in the evaluation of custom QC requirements.

PM's are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to work on a new project, the dissemination of project information and/or project opening meetings may occur to discuss schedules and unique aspects of the project. Items to be discussed may include the project technical profile, turnaround times, holding times, methods, analyte lists, reporting limits, deliverables, sample hazards, or other special requirements. The PM introduces new projects to the laboratory staff through project kick-off meetings or to the supervisory staff during production meetings. These meetings provide direction to the laboratory staff in order to maximize production and client satisfaction, while maintaining quality. In addition, project notes may be associated with each sample batch as a reminder upon sample receipt and analytical processing.

During the project, any change that may occur within an active project is agreed upon between the client/regulatory agency and the PM/laboratory. These changes (e.g., use of a non-standard method or modification of a method) and approvals must be documented prior to implementation. Documentation pertains to any document, e.g., letter, e-mail, variance, contract addendum, which has been signed by both parties.

Such changes are also communicated to the laboratory during operation or supervisor meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

# 7.4 <u>SPECIAL SERVICES</u>

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

**Note:** ISO 17025/NELAC 2003 states that a laboratory "shall afford clients or their representatives cooperation to clarify the client's request". This topic is discussed in Section 7.

The laboratory's standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client's contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

# 7.5 CLIENT COMMUNICATION

Project managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project management will maintain ongoing client communication throughout the entire client project.

Technical Directors are available to discuss any technical questions or concerns that the client may have.

# 7.6 <u>REPORTING</u>

The laboratory works with our clients to produce any special communication reports required by the contract.

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# 7.7 CLIENT SURVEYS

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica's Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

## **SECTION 8**

#### SUBCONTRACTING OF TESTS (NELAC 5.4.5)

## 8.1 <u>OVERVIEW</u>

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase "work sharing" refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in NELAC/ISO 17025 and/or the client's Quality Assurance Project Plan (QAPP). All QC guidelines specific to the client's analytical program are transmitted to the subcontractor and agreed upon before sending the samples to the subcontract facility. Additionally, work requiring accreditation will be placed with an appropriately accredited laboratory. The laboratory performing the subcontracted work will be identified in the final report, as will non-NELAC accredited work where required.

Project Managers (PMs), Customer Service Managers (CSM), or Regional Account Executives (RAE) for the Export Lab are responsible for obtaining client approval prior to outsourcing any samples. The laboratory will advise the client of a subcontract or work sharing arrangement in writing and when possible approval from the client shall be retained in the project folder.

**Note:** In addition to the client, some regulating agencies, such as the US Army Corps of Engineers and the USDA, require notification prior to placing such work.

## 8.2 QUALIFYING AND MONITORING SUBCONTRACTORS

Whenever a PM, Regional Account Executive (RAE), or Customer Service Manager (CSM) becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories and supporting documentation is available on the

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TestAmerica intranet site. Verify necessary accreditation, where applicable, (e.g., on the subcontractors NELAC, A2LA accreditation or State Certification).

- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- NELAC or A2LA accredited laboratories.
- In addition, the firm must hold the appropriate certification to perform the work required.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director/Manager. The Laboratory Director/Manager requests that the QA Manager or Operations Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP No. CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site along with the associate documentation and notify the finance group for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.

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Subcontractors in good standing will be retained on the intranet listing. The QA Manager will
notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any
laboratory requires removal from the intranet site. This notification will be posted on the
intranet site and e-mailed to all Lab Directors/Managers, QA Managers and Sales
Personnel.

# 8.3 OVERSIGHT AND REPORTING

The PM must request that the selected subcontractor be presented with a subcontract, if one is not already executed between the laboratory and the subcontractor. The subcontract must include terms which flow down the requirements of our clients, either in the subcontract itself or through the mechanism of work orders relating to individual projects. A standard subcontract and the Lab Subcontractor Vendor Package (posted on the intranet) can be used to accomplish this, and the Legal & Contracts Director can tailor the document or assist with negotiations, if needed. The PM (or RAE or CSM) responsible for the project must advise and obtain client consent to the subcontract as appropriate, and provide the scope of work to ensure that the proper requirements are made a part of the subcontract and are made known to the subcontractor.

Prior to sending samples to the subcontracted laboratory, the PM confirms their certification status to determine if it's current and scope-inclusive. The information is documented on a Subcontracted Sample Form (Figure 8-1) and the form is retained in the project folder. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a Chain of Custody (COC). A copy of the original COC sent by the client must be included with all samples subbed within TestAmerica.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-NELAC accredited work must be identified in the subcontractor's report as appropriate. If NELAC accreditation is not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

**Note:** The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

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# 8.4 CONTINGENCY PLANNING

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, the QA Manager will be required to verify certifications. The comprehensive approval process must then be initiated within 30 calendar days of subcontracting.

# Figure 8-1. Example - Subcontracting Laboratory Approval Form (Initial / Renewal)

## SUBCONTRACTING LABORATORY APPROVAL

Reference: Section 8 – Quality Assurance Manual

Date: Laboratory: Address:		
Contact and	e-mail address:	

Fax

Contact	and	e-maii	address
Phone:	Dire	ect	

Requested Item <sup>3</sup>	Date Received	Reviewed/ Accepted	Date
1. Copy of State Certification <sup>1</sup>			
2. Insurance Certificate			
3. USDA Soil Permit			
4. Description of Ethics Program <sup>3</sup>			
5. QA Manual <sup>3</sup>			
6. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response <sup>1,3</sup>			
7. State Audit with Corrective Action Response (or NELAC or A2LA Audit) <sup>3</sup>			
8. Sample Report <sup>3</sup>			
9. SOQ or Summary list of Technical Staff and Qualifications <sup>3</sup>			
10. SOPs for Methods to Be Loadshifted <sup>2,3</sup>			
11. For DoD Work: Statement that Lab quality system complies with QSM.			
12. For DoD Work: Approved by specific DoD Component laboratory approval process.			

1 - Required when emergency procedures are implemented.

2 - Some labs may not submit copies due to internal policies. In these cases, a copy of the first page and signature page of the SOP is acceptable. This requirement may also be fulfilled by supplying a table of SOPs with effective dates.

3 – If the laboratory has NELAC accreditation, Item #s 4 through 10 are not required.

On Site Audit Planned: YES	NO I	f yes, Date Completed	l:	By Whom:
Comments:				
Lab Acceptable for Subcontract	ting Work:	: YES NO	Limitations:	
QA Manager:(Prir	nted Name		Date:	
□ Forwarded to Contract Coord	dinator, by	y:		_ Date:

## SECTION 9

## PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

# 9.1 <u>OVERVIEW</u>

Evaluation and selection of suppliers and vendors is performed, in part, on the basis of the quality of their products, their ability to meet the demand for their products on a continuous and short term basis, the overall quality of their services, their past history, and competitive pricing. This is achieved through evaluation of objective evidence of quality furnished by the supplier, which can include certificates of analysis, recommendations, and proof of historical compliance with similar programs for other clients. To ensure that quality critical consumables and equipment conform to specified requirements, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

# 9.2 <u>GLASSWARE</u>

Glassware used for volumetric measurements must be Class A or verified for accuracy according to laboratory procedure. Pyrex (or equivalent) glass should be used where possible. For safety purposes, thick-wall glassware should be used where available.

# 9.3 REAGENTS, STANDARDS & SUPPLIES

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pretested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

## 9.3.1 <u>Purchasing</u>

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

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The laboratory utilizes the JD Edwards One World software accessed thru the corporate intranet for materials requisitions. User names and passwords are distributed to authorized personnel. Orders are placed bi-weekly by the users and are approved by the Laboratory Director. Only corporate approved suppliers are allowed to be used.

Orders are reviewed by Corporate and placed to the suppliers.

The analyst may also check the item out of the on-site consignment system that contains items approved for laboratory use.

# 9.3.2 <u>Receiving</u>

It is the responsibility of the purchasing manager to receive the shipment. It is the responsibility of the analyst who ordered the materials to date the material when received. Once the ordered reagents or materials are received, the analyst compares the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals.

## 9.3.3 <u>Specifications</u>

All methods in use in the laboratory specify the grade of reagent that must be used in the procedure. If the quality of the reagent is not specified, it may be assumed that it is not significant in that procedure and, therefore, any grade reagent may be used. It is the responsibility of the analyst to check the procedure carefully for the suitability of grade of reagent.

Chemicals must not be used past the manufacturer's expiration date and must not be used past the expiration time noted in a method SOP. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals unless noted otherwise by the manufacturer or by the reference source method. Chemicals should not be used past the manufacturer's or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date can not be extended if the dry chemical is discolored or appears otherwise physically degraded, the dry chemical must be discarded.
- Expiration dates can be extended if the dry chemical is found to be satisfactory based on acceptable performance of quality control samples (Continuing Calibration Verification (CCV), Blanks, Laboratory Control Sample (LCS), etc.).
- If the dry chemical is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical is compared to an unexpired independent source in performing the method and the performance of the dry chemical is found to be satisfactory.

The comparison must show that the dry chemical meets CCV limits. The comparison studies are maintained with the QA Manager.

Wherever possible, standards must be traceable to national or international standards of measurement or to national or international reference materials. Records to that effect are available to the user.

Compressed gases in use are checked for pressure and secure positioning daily. The minimum total pressure must be 500 psig or the tank must be replaced. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1- mmho/cm (or specific resistivity of greater than 1.0 megaohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Department Managers/Supervisors must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased VOA vials must be certified clean and the certificates must be maintained. If uncertified VOA vials are purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable). Incorporation of the item into the record indicates that the analyst has compared the new certificate with the previous one for the same purpose and that no difference is noted, unless approved and so documented by the Technical Director or QA Manager.

# 9.3.4 <u>Storage</u>

Reagent and chemical storage is important from the aspects of both integrity and safety. Lightsensitive reagents may be stored in brown-glass containers. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

## 9.4 PURCHASE OF EQUIPMENT/INSTRUMENTS/SOFTWARE

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate

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Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department or QA Department. Software certificates supplied by the vendors are filed with the LIMS Administrator. The manufacturer's operation manual is retained at the bench.

# 9.5 <u>SERVICES</u>

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Department Managers. The service providers that perform the services are approved by the Department Managers and Laboratory Director.

## 9.6 <u>SUPPLIERS</u>

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

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The laboratory has access to a listing of all approved suppliers of critical consumables, supplies and services. This information is provided through the JD Edwards purchasing system.

## 9.6.1 <u>New Vendor Procedure</u>

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form.

New vendors are evaluated based upon criteria appropriate to the products or services provided as well as their ability to provide those products and services at a competitive cost. Vendors are also evaluated to determine if there are ethical reasons or potential conflicts of interest with TestAmerica employees that would make it prohibitive to do business with them as well as their financial stability. The QA Department and/or the Laboratory Director are consulted with vendor and product selection that have an impact on quality.

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## **SECTION 10**

#### COMPLAINTS (NELAC 5.4.8)

## 10.1 <u>OVERVIEW</u>

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and is documented following SOP CT-MKS-7. Complaints are documented and tracked utilizing the lims Non-Conformance module. An NCM is generated within the lims system and the complaint is fully documented and connected with the job for which it originated from. At the end of each month, all complaints are compiled using a the Management reports in LIMS and all are listed in the monthly QA report.

#### 10.2 EXTERNAL COMPLAINTS

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to (SOP# CT-MKS-7).

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelyhood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery

• Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

# 10.3 INTERNAL COMPLAINTS

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

# 10.4 MANAGEMENT REVIEW

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

# **SECTION 11**

## CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

# 11.1 <u>OVERVIEW</u>

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed.

When an analyst encounters such a situation, the problem is presented to the supervisor for resolution. The supervisor may elect to discuss it with the Technical Director or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it using the laboratories corrective action system described in Section 12. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Laboratory Director and QA Manager, documented and included in the project folder. Deviations **must** also be noted on the final report with a statement that the compound is not reported in compliance with NELAC (or the analytical method) requirements and the reason. Data being reported to a non-NELAC state would need to note the change made to how the method is normally run.

# 11.2 **RESPONSIBILITIES AND AUTHORITIES**

TestAmerica's Corporate SOP entitled *Internal Investigation of Potential Data Discrepancies and Determination for Data Recall* (SOP No. CA-L-S-001), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director or QA Manager may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient

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sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised\_of the Laboratory Director, the QA Manager, and the Department Managers. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures <u>must</u> be conveyed to an Ethics and Compliance Officer (ECO), Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director/Manager, QA Manager, ECOs, Corporate Quality, the COO, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work.

# 11.3 EVALUATION OF SIGNIFICANCE AND ACTIONS TAKEN

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CA-L-S-001) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CA-L-S-001.

# 11.4 PREVENTION OF NONCONFORMING WORK

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. On a monthly basis, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

# 11.5 METHOD SUSPENSION/RESTRICTION (STOP WORK PROCEDURES)

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical Director, QA Manager, Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

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# **SECTION 12**

## CORRECTIVE ACTION (NELAC 5.4.10)

# 12.1 <u>OVERVIEW</u>

A major component of TestAmerica's Quality Assurance (QA) Program is the problem investigation and feedback mechanism designed to keep the laboratory staff informed on quality related issues and to provide insight to problem resolution. When nonconforming work or departures from policies and procedures in the quality system or technical operations are identified, the corrective action procedure provides a systematic approach to assess the issues, restore the laboratory's system integrity, and prevent reoccurrence. Corrective actions are documented using Non-Conformance Memo's (NCM) and Corrective Action Reports (CAR) (refer to Figure 12-1 and 12-2).

# 12.2 <u>GENERAL</u>

Problems within the quality system or within analytical operations may be discovered in a variety of ways, such as QC sample failures, internal or external audits, proficiency testing (PT) performance, client complaints, staff observation, etc..

The purpose of a corrective action system is to:

- Identify non-conformance events and assign responsibility(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify Systematic Problems before they become serious.
- Identify and track client complaints and provide resolution.

**12.2.1** <u>Non-Conformance Memo (NCM)</u> - is used to document the following types of corrective actions:

- Deviations from an established procedure or SOP
- QC outside of limits (non-matrix related)
- Isolated reporting / calculation errors
- Client complaints

**12.2.2** <u>Corrective Action Report (CAR)</u> - is used to document the following types of corrective actions:

- Questionable trends that are found in the monthly review of NCMs.
- Issues found while reviewing NCMs that warrant further investigation.
- Internal and external audit findings.
- Failed or unacceptable PT results.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors

## **Company Confidential & Proprietary**

- Health and Safety violations
- •

# 12.3 CLOSED LOOP CORRECTIVE ACTION PROCESS

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

# 12.3.1 <u>Cause Analysis</u>

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM or CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Supervisor, Laboratory Director/Manager, or QA Manager (or QA designee) is consulted.

# 12.3.2 Selection and Implementation of Corrective Actions

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The NCM or CAR is used for this documentation.

# 12.3.3 Monitoring of the Corrective Actions

- The Department Manager/Supervisor and QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- Each NCM is generated through the LIMS system. NCM are tracked and a monthly summary of all corrective actions can be generated and exported to excel for review to aid in esuring that the corrective actions have taken effect.
- The QA Manager reviews monthly NCMs and CARs for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the outof-control situation and problems encountered in solving the situation.

# 12.3.4 Follow-up Audits

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.2.4, Special Audits.)

## 12.4 TECHNICAL CORRECTIVE ACTIONS

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of an NCM or CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintains Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions, QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where sample results may be impaired, the Project Manager is notified by an NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

## 12.5 BASIC CORRECTIONS

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original "uncorrected" file must be maintained intact and a second "corrected" file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

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When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

# Figure 12-1. Example - Corrective Action Report

	LEADER IN ENVIRONME		Corrective	Action Report	Page 1 of 1
		Open Date:	. <u>.</u>	Initiated By:	
<u>Basis</u>					
0	Audit		Complaint		
D	PT Failure SOP Departure	а 0	QC Failure Prevention		
Desc	ription:				
Meth					
Root	Cause / Purpose:				
		Investigated By	7 <b>-</b>	D	ite:
				~~	
Poter	ntial Corrective / Pr	eventive Actions:	•		
		Decomposited D.	**	D	ite:
L		Recommended By	rz	<i>D</i> :	
Corr	ective Actions Perfo	ormed:			
		Performed By	<u>/:</u>	D:	ate:
Disp □	osition of Data: Reanalyzed				
0	Rejected				
۵	Recalled				
Follo	w-Up Activities:				
*****					
	ontinue with anothe hange SOP	r Corrective Acti Assessed B		D;	ite:
	Manager:		Date:	Closed D:	ste <sup>*</sup>
L Ya I	uameger:		J MARC.	CIUSTS DA	

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# Figure 12-2 Example – Non-Conformance Memo

NCM ID: 2555 Lab Section: Gas Chromatography Semi-V NCM Type: Reporting Limit - Dilution. Ma NCM Category: Anomaly		Status: Approved CreatedBy: Passarell		
Narrative Internal Comments		Affected Items		
		+Add Flemov	2	
in the sample: <&commaMerge&>. Elevate	and up for sulfur due to the extremely high amount of sulfur ed reporting limits (RLs) are provided for Heplachlor, as this ilysis. A straight analysis has been reported for all or.	Pessipi		Final Report
Detail/History		Notifications		
tt User Name Entry Date	BZUA BEEKS	+Add Remov	s	
1         Passarella, Daniel         9/19/2007 1           2         Passarella, Daniel         9/19/2007 1	re-link	Use: Name May, Davin M	Notice Level Level 1	Veilfoation Typ Review
3 May, Dawn M 9/19/2007 1:	The following sample was diluted and cleaned up for sufur due to the extremely high amount of sufur in the sample: <&commakereak>. Elevated reporting limits (RLs) are provided.	Culk, Marsha	Level 1	Review

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# Table 12-1.

# **Example – General Corrective Action Procedures**

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Initial Instrument Blank <i>(Analyst)</i>	- Instrument response < ½ RL.	<ul> <li>Prepare another blank.</li> <li>If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc</li> </ul>
Initial Calibration Standards (Analyst, Supervisor)	<ul> <li>Correlation coefficient &gt; 0.99 or standard concentration value.</li> <li>% Recovery/%RSD within acceptance range.</li> <li>See details in Method SOP.</li> </ul>	<ul> <li>Reanalyze standards.</li> <li>If still unacceptable, remake standards and/or recalibrate instrument.</li> </ul>
Independent Calibration Verification (Second Source) <i>(Analyst, Supervisor)</i>	- % Recovery within control limits.	<ul> <li>Remake and reanalyze standard.</li> <li>If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.</li> </ul>
Continuing Calibration Standards (Analyst, Data Reviewer)	% Recovery/ % Difference within control limits.	<ul> <li>Reanalyze standard.</li> <li>If still unacceptable, then recalibrate and rerun affected samples.</li> </ul>
Matrix Spike / Matrix Spike Duplicate (MS/MSD) <i>(Analyst, Data Reviewer)</i>	- % Recovery within limits documented in SOPs/LIMS	<ul> <li>If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS.</li> <li>If the LCS is within acceptable limits the batch is acceptable.</li> <li>The results of the duplicates, matrix spikes and the LCS are reported with the data set.</li> </ul>
Laboratory Control Sample (LCS) (Analyst, Data Reviewer)	- % Recovery within limits specified in SOPs/LIMS.	<ul> <li>Batch must be re-prepared and re- analyzed.</li> <li>Note: If there is insufficient sample or the holding time cannot be met, contact client and report with flags.</li> </ul>
Surrogates (Analyst, Data Reviewer)	- % Recovery within limits of method /Standard Operating Procedures	- Individual sample must be repeated/reextracted. Place comment in LIMS.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Method Blank (MB_ (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	<ul> <li>Reanalyze blank.</li> <li>If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.</li> </ul>
Proficiency Testing (PT) Samples (QA Manager, Department Manager/Supervisor)	- Criteria supplied by PT Supplier.	- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.
Reporting / Calculation Errors (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Department Manager/ Supervisor, QA Manager, Corporate QA, Corporate Management)	- SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CA-L-S-001.
Client Complaints (Project Managers, Lab Director/Manager, Sales and Marketing)	-	- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow- up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report (Refer to Section 17 for an example) (QA Manager, Lab Director/Manager, Department Supervisors/Managers)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.

#### Note:

1. Except as noted below for certain compounds, the method blank should be below the detection limit. Certain programs may require less than ½ the detection limit. Concentrations up to five times the reporting limit will be allowed for the ubiquitous laboratory and reagent contaminants: methylene chloride, acetone, 2-butanone and phthalates. This allowance presumes that the detection limit is significantly below any regulatory limit to which the data are to be compared and that blank subtraction will not occur.

## **SECTION 13**

## PREVENTIVE ACTION (NELAC 5.4.11)

# 13.1 <u>OVERVIEW</u>

The laboratory's preventive action programs improve, or eliminate potential causes of nonconforming product and/or nonconformance to the quality system. This preventive action process is a proactive continuous process improvement activity that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the QA Metrics Report, internal or external audits, proficiency testing performance, client complaints, staff observation, etc..

The monthly QA Metrics Report shows performance indicators in all areas of the quality system. These areas include revised reports, corrective actions, audit findings, internal auditing and data authenticity audits, client complaints, PT samples, holding time violations, SOPs, ethics training, etc. These metrics are used to help evaluate quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system:

- <u>Identification</u> of an opportunity for preventive action.
- <u>Process</u> for the preventive action.
- Define the measurements of the effectiveness of the process once undertaken.
- <u>Execution</u> of the preventive action.
- Evaluation of the plan using the defined measurements.
- Verification of the effectiveness of the preventive action.
- <u>Close-Out</u> by documenting any permanent changes to the Quality System as a result of the Preventive Action. Documentation of Preventive Action is incorporated into the monthly QA reports, corrective action process and management review.

**13.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the Annual Management Review (Section 16). A highly detailed recap is not required; a simple

recount of success and failure within the preventive action program will provide management a measure for evaluation.

# 13.2 MANAGEMENT OF CHANGE

The Management of Change process is designed to manage significant events and changes that occur within the laboratory. Through these various tracking indicators, the potential risks inherent with a new event or change are identified and evaluated. The risks are minimized or eliminated through pre-planning and the development of preventive measures. The types of indicators monitored under this collective system include:

- SOP Tracking
  - Current Revisions w/ Effective Dates
  - Required Biennial Revisions w/ Due Date
- Proficiency Testing (PT) Sample Tracking
  - Pass / Fail most current 2 out of 3 studies.
- Instrument / Equipment List
  - Current / Location
- Accreditations
  - o New / Expiring
- Method Capabilities
  - Current Listing by program (e.g., Potable Water, Soils, etc.)
- Key Personnel
  - o Technical Managers, Department Supervisors, etc..

These items are maintained on TestAmerica's Intranet (Proposal Library) or on our internal database (TotalAccess) which uploads to our company internet site.

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## **SECTION 14**

## CONTROL OF RECORDS (NELAC 5.4.12)

The laboratory maintains a record system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

# 14.1 <u>OVERVIEW</u>

The laboratory has established procedures for identification, collection, indexing, access, filing, storage, maintenance and disposal of quality and technical records. A record index is listed in Table 14-1. Quality records are maintained by the QA department in a database, which is backed up as part of the regular laboratory backup or archived in banker boxes. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by department supervisors.

	Record Types <sup>1</sup> :	Retention Time:
Technical Records	- Raw Data - Logbooks <sup>2</sup> - Standards - Certificates - Analytical Records - Lab Reports	5 Years from analytical report issue*
Official Documents	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Manuals	5 Years from document retirement date*
QA Records	<ul> <li>Internal &amp; External Audits/Respones</li> <li>Certifications</li> <li>Corrective/Preventive Actions</li> <li>Management Reviews</li> <li>Method &amp; Software Validation / Verification Data</li> <li>Data Investigation</li> </ul>	5 Years from archival* <u><b>Data Investigation:</b></u> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
Project Records	<ul> <li>Sample Receipt &amp; COC</li> <li>Documentation</li> <li>Contracts and Amendments</li> <li>Correspondence</li> <li>QAPP</li> <li>SAP</li> <li>Telephone Logbooks</li> <li>Lab Reports</li> </ul>	5 Years from analytical report issue*

## Table 14-1. Record Index<sup>1</sup>

	Record Types <sup>1</sup> :	Retention Time:
Administrative Records	Finance and Accounting	10 years
	EH&S Manual, Permits, Disposal Records	7 years
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

<sup>1</sup> Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility or an offsite location that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees. Records archived off-site are stored in a secure location. Whether on-site or off-site storage is used, logs are maintained in each storage box to note removal and return of records. Retention of records are maintained on-site at the laboratory for at least 1 year after their generation and moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

# 14.1.2 Programs with Longer Retention Requirements

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

Program	<sup>1</sup> Retention Requirement
Drinking Water – All States	10 years (project records)
Commonwealth of MA – All environmental data 310 CMR 42.14	10 years
NY Potable Water NYCRR Part 55-2	10 years

# Table 14-2. Example: Special Record Retention Requirements

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**14.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.12.1 for more information. More Information on data archive can be found in the SOP for Data Backup Procedure, CT-SYS-31.

**14.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data (Records stored off site should be accessible within 2 days of a request for such records). The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler. If any sampling notes are provided with a work order, they are kept with this package.
- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set. Instrument data is stored sequentially by instrument. A given day's analyses are maintained in the order of the analysis. Run logs are maintained for each instrument or method; a copy of each day's run long or instrument sequence is stored with the data to aid in re-constructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP CT-RPS-7, Lims final Reporting.
- Also refer to Section 19.13.1 'Computer and Electronic Data Related Requirements'.

# 14.2 TECHNICAL AND ANALYTICAL RECORDS

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for the performance of each analysis and reviewing results.

**14.2.2** Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**14.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; Time of Analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a benchsheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically stored in method parameters associated with each data file, where available.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- sample preparation including cleanup, separation protocols, incubation periods or

subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;

- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

# 14.3 LABORATORY SUPPORT ACTIVITIES

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;
- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

# 14.3.1 <u>Sample Handling Records</u>

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and

• procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

# 14.4 ADMINISTRATIVE RECORDS

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

# 14.5 RECORDS MANAGEMENT, STORAGE AND DISPOSAL

**14.5.1** All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

**14.5.2** All information necessary for the historical reconstruction of data is maintained by the laboratory. Records that are stored only on electronic media must be supported by the hardware and software necessary for their retrieval.

**14.5.3** Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

**14.5.4** The laboratory has a record management system (a.k.a. as document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS – no logbooks are used to record that data. Records are considered archived when noted as such in the records management system (a.k.a. as document control).

# 14.5.5 <u>Transfer of Ownership</u>

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

# 14.5.6 <u>Records Disposal</u>

**14.5.6.1** Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

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**14.5.6.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

**14.5.6.3** If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

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#### **SECTION 15**

#### AUDITS (NELAC 5.4.13)

#### 15.1 INTERNAL AUDITS

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and when requested to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Audits, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are shown in Table 16-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

Description	Performed by	Frequency
Quality Systems	QA Department or Designee	All areas of the laboratory annually
QA Technical Audits - Evaluate raw data versus final reports - Analyst integrity - Data authenticity	QA Department or Designee	All methods within a 2-year period, with at least 15% of methods every quarter
SOP Method Compliance	Technical Director	<ul> <li>All SOPs within a 2-year period</li> <li>All new analysts or new analyst/methods within 3 months of IDOC</li> </ul>
Special	QA Department or Designee	Surveillance or spot checks performed as needed
Performance Testing	Analysts with QA oversight	Two successful per year for each NELAC field of testing or as dictated by regulatory requirements

Table 15-1.	Types	of Internal	Audits and	Frequency
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#### 15.1.1 Annual Quality Systems Audit

An annual quality systems audit is required to ensure compliance to analytical methods and SOPs, the laboratory's Data Integrity and Ethics Policies, NELAC quality systems, client and state requirements, and the effectiveness of the internal controls of the analytical process, including but not limited to data review, quality controls, preventive action and corrective action. The completeness of earlier corrective actions is assessed. The audit is divided into modules for each operating or support area of the lab, and each module is comprehensive for a given

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area. The area audits may be done on a rotating schedule throughout the year to ensure adequate coverage of all areas. This schedule may change as situations in the laboratory warrant.

# 15.1.2 **QA Technical Audits**

QA technical audits are based on client projects, associated sample delivery groups, and the methods performed. Reported results are compared to raw data to verify the authenticity of results. The validity of calibrations and QC results are compared to data qualifiers, footnotes, and case narratives. Documentation is assessed by examining run logs and records of manual integrations. Manual calculations are checked. Where possible, MintMiner is used to identify unusual manipulations of the data deserving closer scrutiny. QA technical audits will include all methods within a two-year period.

# 15.1.3 <u>SOP Method Compliance</u>

Compliance of all SOPs with the source methods and compliance of the operational groups with the SOPs will be assessed by the Technical Director at least every two years. The work of each newly hired analyst is assessed within 3 months of working independently, (e.g., completion of method IDOC). In addition, as analysts add methods to their capabilities, (new IDOC) reviews of the analyst work products will be performed within 3 months of completing the documented training.

### 15.1.4 Special Audits

Special audits are conducted on an as needed basis, generally as a follow up to specific issues such as client complaints, corrective actions, PT results, data audits, system audits, validation comments, regulatory audits or suspected ethical improprieties. Special audits are focused on a specific issue, and report format, distribution, and timeframes are designed to address the nature of the issue.

#### 15.1.5 <u>Performance Testing</u>

The laboratory participates semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Drinking Water, Nonpotable Water, and Soil.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

# 15.2 EXTERNAL AUDITS

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

# 15.2.1 <u>Confidential Business Information (CBI) Considerations</u>

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or "company confidential". Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2003 NELAC standards.

# 15.3 <u>AUDIT FINDINGS</u>

Audit findings are documented using the corrective action process and database. The laboratory's corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. A copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory's test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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Clients must be notified promptly in writing, of any event such as the identification of defective measuring or test equipment that casts doubt on the validity of results given in any test report or amendment to a test report. The investigation must begin within 24-hours of discovery of the problem and all efforts are made to notify the client within two weeks after the completion of the investigation.

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### **SECTION 16**

### MANAGEMENT REVIEWS (NELAC 5.4.14)

# 16.1 QUALITY ASSURANCE REPORT

A comprehensive QA Report shall be prepared each month by the laboratory's QA Department and forwarded to the Laboratory Director, Technical Directors, Operation Manager, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Senior Management Team and General Managers.

# 16.2 <u>ANNUAL MANAGEMENT REVIEW</u>

The senior lab management team (Laboratory Director, Technical Directors, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining quality goals & objectives. Corporate Operations and Corporate QA personnel is be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management review (Corporate Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the "big picture" by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:

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- Adequacy of staff, equipment and facility resources.
- Adequacy of policies and procedures.
- Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

#### 16.3 POTENTIAL INTEGRITY RELATED MANAGERIAL REVIEWS

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CA-L-S-001). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's COO, VP of Client & Technical Services, General Managers and Quality Directors receive a monthly report from the Director of Quality & Client Advocacy summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

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#### **SECTION 17**

#### PERSONNEL (NELAC 5.5.2)

#### 17.1 <u>OVERVIEW</u>

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

#### 17.2 <u>EDUCATION AND EXPERIENCE REQUIREMENTS FOR TECHNICAL</u> <u>PERSONNEL</u>

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. Selection of qualified candidates for laboratory employment begins with documentation of minimum education, training, and experience prerequisites needed to perform the prescribed task. Minimum education and training requirements for TestAmerica employees are outlined in job descriptions and are generally summarized for analytical staff in the table below.

The laboratory maintains job descriptions for all personnel who manage, perform or verify work affecting the quality of the environmental testing the laboratory performs. Job Descriptions are located on the TestAmerica intranet site's Human Resources web-page (Also see Section 4 for position descriptions/responsibilities).

Experience and specialized training are occasionally accepted in lieu of a college degree (basic lab skills such as using a balance, aseptic or quantitation techniques, etc., are also considered).

As a general rule for analytical staff:

Specialty	Education	Experience	
Extractions, Digestions, some electrode methods (pH, DO, Redox, etc.), or Titrimetric and Gravimetric Analyses	H.S. Diploma	On the job training (OJT)	
CVAA, Single component or short list Chromatography (e.g., Fuels, BTEX-GC, IC	A college degree in an applied science or 2 years of college and at least 1 year of college chemistry	Or 2 years prior analytical experience is required	
ICP, ICPMS, Long List or complex chromatography (e.g., Pesticides, PCB, Herbicides, HPLC, etc.), GCMS	A college degree in an applied science or 2 years of college chemistry	or 5 years of prior analytical experience	
Spectra Interpretation	A college degree in an applied science or 2 years of college chemistry	And 2 years relevant experience Or 5 years of prior analytical experience	
Technical Directors/Department Managers – <u>General</u>	Bachelors Degree in an applied science or engineering with 24 semester hours in chemistry An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years experience in environmental analysis of representative analytes for which they will oversee	
Technical Director – <u>Wet Chem</u> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience	

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Department Manager, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

# 17.3 <u>TRAINING</u>

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Below are examples of various areas of required employee training:

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All
Ethics – Comprehensive Refresher	Annually	All
Initial Demonstration of Capability (DOC)	Prior to unsupervised method performance	Technical

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to "Demonstration of Capability" in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee's secured personnel file.

Further details of the laboratory's training program are described in the Employee Training SOP (CT-QAS-16).

# 17.4 DATA INTEGRITY AND ETHICS TRAINING PROGRAM

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation within 1 week of hire, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

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In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CA-L-P-001) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

### **SECTION 18**

#### ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

### 18.1 <u>OVERVIEW</u>

The laboratory is a 14,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, and administrative functions.

# 18.2 <u>ENVIRONMENT</u>

Laboratory accommodation, test areas, energy sources, lighting are adequate to facilitate proper performance of tests. The facility is equipped with heating, ventilation, and air conditioning (HVAC) systems appropriate to the needs of environmental testing performed at this laboratory.

The environment in which these activities are undertaken does not invalidate the results or adversely affect the required accuracy of any measurements.

The laboratory provides for the effective monitoring, control and recording of environmental conditions that may effect the results of environmental tests as required by the relevant specifications, methods, and procedures. Such environmental conditions include humidity, voltage, temperature, and vibration levels in the laboratory.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

# 18.3 WORK AREAS

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

 Volatile organic chemical handling areas, including sample preparation and waste disposal, and volatile organic chemical analysis areas. The volatile analysis laboratory containing GC/MS instrumentation has a separate air handling system which is maintained at a positive pressure at all times. The organic sample preparation laboratory has a separate HVAC system that creates negative pressure in the area. This design results in a contaminant-free environment for trace level volatile analysis.

Access to and use of all areas affecting the quality of analytical testing is defined and controlled by secure access to the laboratory building as described below in the Building Security section.

Adequate measures are taken to ensure good housekeeping in the laboratory and to ensure that any contamination does not adversely affect data quality. These measures include regular cleaning to control dirt and dust within the laboratory.

Work areas are available to ensure an unencumbered work area. Work areas include:

- Access and entryways to the laboratory.
- Sample receipt areas.
- Sample storage areas.
- Chemical and waste storage areas.
- Data handling and storage areas.
- Sample processing areas.
- Sample analysis areas.

# 18.4 FLOOR PLAN

A floor plan can be found in Appendix 1.

### 18.5 BUILDING SECURITY

Building keys are distributed to employees as necessary.

Visitors to the laboratory sign in and out in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contains requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook.

### SECTION 19

#### TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

### 19.1 <u>OVERVIEW</u>

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

### 19.2 STANDARD OPERATING PROCEDURES (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled in the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for writing an SOP are incorporated by reference to TestAmerica's Corporate SOP entitled 'Writing a Standard Operating Procedure', No. CW-Q-S-002 or the laboratory's SOP entitled 'SOP for Generating SOPS, #CT-QAS-8.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water and DoD SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

# 19.3 LABORATORY METHODS MANUAL

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

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The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

# 19.4 <u>SELECTION OF METHODS</u>

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

# 19.4.1 <u>Sources of Methods</u>

Routine analytical services are performed using standard EPA-approved methodology. In some cases, modification of standard approved methods may be necessary to provide accurate analyses of particularly complex matrices. When the use of specific methods for sample analysis is mandated through project or regulatory requirements, only those methods shall be used.

When clients do not specify the method to be used or methods are not required, the methods used will be clearly validated and documented in an SOP and available to clients and/or the end user of the data.

The analytical methods used by the laboratory are those currently accepted and approved by the U. S. EPA and the state or territory from which the samples were collected. Reference methods include:

- <u>Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act</u>, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. <u>Revised as of July 1, 1995</u>, <u>Appendix</u> <u>A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)</u>
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- <u>Methods for the Determination of Inorganic Substances in Environmental Samples</u>, EPA-600/R-93/100, August 1993.
- <u>Methods for the Determination of Metals in Environmental Samples</u>, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- <u>Methods for the Determination of Organic Compounds in Drinking Water</u>, EPA-600/4-88-039, December 1988, Revised, July 1991, Supplement I, EPA-600-4-90-020, July 1990, Supplement II, EPA-600/R-92-129, August 1992. <u>Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series)</u> (EPA 500 Series methods)
- <u>Technical Notes on Drinking Water Methods</u>, EPA-600/R94-173, October 1994
- <u>NIOSH Manual of Analytical Methods</u>, 4<sup>th</sup> ed., August 1994.

- <u>Statement of Work for Inorganics Analysis</u>, ILM04.1, USEPA Contract Laboratory Program Multimedia, Multi-concentration.
- <u>Statement of Work for Organics Analysis</u>, OLM04.2, USEPA Contract Laboratory Program, Multimedia, Multi-concentration.
- <u>Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration, OLMO4.1, USEPA</u> Contract Laboratory Program, September 1998.
- <u>Standard Methods for the Examination of Water and Wastewater</u>, 18<sup>th</sup>/19<sup>th</sup>/20<sup>th</sup> edition; Eaton, A.D. Clesceri, L.S. Greenberg, A.E. Eds; American Water Works Association, Water Pollution Control Federation, American Public Health Association: Washington, D.C.
- <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- <u>Annual Book of ASTM Standards</u>, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### 19.4.2 Demonstration of Capability

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

**19.4.2.1** A demonstration of capability (Demonstration of Capability(DOC), Lab SOP # CT-RPS-7) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel.

**19.4.2.2** The initial demonstration of capability must be thoroughly documented and approved by the Technical Director/Department Manager and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures.

**19.4.2.3** The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL).
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*

# 19.4.3 Initial Demonstration of Capability (IDOC) Procedures

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem and repeat the test for all parameters of interest beginning with 19.4.3.3 above.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

A certification statement (refer to Figure 19-1 as an example) shall be used to document the completion of each initial demonstration of capability. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples must have been compared to the laboratory's quality control acceptance limits.

# 19.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

# 19.6 VALIDATION OF METHODS

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

# 19.6.1 <u>Method Validation and Verification Activities for All New Methods</u>

While method validation can take various courses, the following activities can be required as part of method validation. Method validation records are designated QC records and are archived accordingly.

# 19.6.1.1 Determination of Method Selectivity

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Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

# 19.6.1.2 Determination of Method Sensitivity

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

# 19.6.1.3 <u>Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)</u>

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

# 19.6.1.4 Determination of Interferences

A determination that the method is free from interferences in a blank matrix is performed.

# 19.6.1.5 Determination of Range

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### 19.6.1.6 Determination of Accuracy and Precision

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### 19.6.1.7 Documentation of Method

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The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

# 19.6.1.8 Continued Demonstration of Method Performance

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

### 19.7 METHOD DETECTION LIMITS (MDL)/ LIMITS OF DETECTION (LOD)

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally, the analyst prepares at least seven replicates of solution spiked at one to five times the estimated method detection limit (most often at the lowest standard in the calibration curve) into the applicable matrix with all the analytes of interest. Each of these aliquots is extracted (including any applicable clean-up procedures) and analyzed in the same manner as the samples. Where possible, the seven replicates should be analyzed over 2-4 days to provide a more realistic MDL. To allow for some flexibility, this low level standard may be analyzed every batch or every week or some other frequency rather than doing the study all at once. In addition, a larger number of data points may be used if the appropriate t-value multiplier is used

Refer to the Corporate SOP No. CA-Q-S-006 or the laboratory's SOP No. CT-QAS-17 for details on the laboratory's MDL process.

### 19.8 INSTRUMENT DETECTION LIMITS (IDL)

**19.8.1** The IDL is sometimes used to assess the reasonableness of the MDLs or in some cases required by the analytical method or program requirements. IDLs are most used in metals analyses but may be useful in demonstration of instrument performance in other areas.

**19.8.2** IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 x the absolute value of the standard deviation.

**19.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

# 19.9 VERIFICATION OF DETECTION AND REPORTING LIMITS

**19.9.1** Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at approximately 2-3 times the calculated MDL

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for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and 1-4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will not report to the MDL, or redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually.

**19.9.2** When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1-2 times the reporting limit and annually thereafter. The annual requirement is waved for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements.

# 19.10 RETENTION TIME WINDOWS

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specific in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte's retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

# 19.11 EVALUATION OF SELECTIVITY

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption and specific electrode response factors.

# 19.12 ESTIMATION OF UNCERTAINTY OF MEASUREMENT

**19.12.1** Uncertainty is "a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand" (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result's validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an "expanded uncertainty": the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor k=2.

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable,

assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as 1.0 + 0.5 mg/l.

**19.12.5** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

# 19.13 SAMPLE REANALYSIS GUIDELINES

Because there is a certain level of uncertainty with any analytical measurement, a sample reanalysis may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supercede the following items.** 

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within <u>+</u> 1 reporting limit for samples <u><</u> 5x the reporting limit, the original analysis will be reported. At the client's request, both results may be reported on the same report but not on two separate reports.
- If the reanalysis does not agree (as defined above) with the original result, then the laboratory will investigate the discrepancy and reanalyze the sample a third time for confirmation if sufficient sample is available.
- Any potential charges related to reanalysis are discussed in the contract terms and conditions or discussed at the time of the request. The client will typically be charged for reanalysis unless it is determined that the lab was in error.

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 Due to the potential for increased variability, reanalysis may not be applicable to Nonhomogenous, Encore, and Sodium Bisulfate preserved samples. See the Area Supervisor or Laboratory Director/Manager if unsure.

# 19.14 CONTROL OF DATA

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

### 19.14.1 Computer and Electronic Data Related Requirements

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOPs for back-up recovery and archive for each of the servers. The laboratory is currently running the TALS Lims system which is a custom in-house developed LIMS system that has been highly customized to meet the needs of the laboratory. It is referred to as LIMS for the remainder of this section. The LIMS utilizes Microsoft SQL Server which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

- **19.14.1.1** <u>Maintain the Database Integrity:</u> Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.
  - LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.
  - Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use.
- **19.14.1.2** <u>Ensure Information Availability:</u> Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.
- **19.14.1.3** <u>Maintain Confidentiality:</u> Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

# 19.14.2 Data Reduction

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Manual integration of peaks will be documented and reviewed and the raw data will be flagged in accordance with the TestAmerica Corporate SOP No. CA-Q-S-002, *Acceptable Manual Integration Practices*.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

- **19.14.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/<u>year</u>). It must be easily identifiable who performed which tasks if multiple people were involved.
- **19.14.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ( $\mu$ g/l) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ( $\mu$ g/kg) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.
- **19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.
- **19.14.2.4** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.
- **19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

#### 19.14.3 Logbook / Worksheet Use Guidelines

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample

ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be "Z"'d out, signed and dated.
- Worksheets are created with the approval of the Department Manager and QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

# 19.14.4 <u>Review / Verification Procedures</u>

Review procedures are out lined in several SOPs [e.g. Sample Control,, Project Management] and work instructions, to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported. The laboratory also has a corporate SOP discussing Manual Integrations to ensure the authenticity of the data CA-Q-S-002 as well as information within the analytical method SOPs discussing the reason code documentation. The general review concepts are discussed below, more specific information can be found in the SOPs.

- **19.14.4.1** The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into a computer LIMS. The Sample Control Supervisor reviews the transaction of the chain-of-custody forms and the inputted information. The Project Managers perform final review of the chain-of-custody forms and inputted information.
- **19.14.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant EPA methodologies. The Analysts transfer the data into the LIMS and add data qualifiers if applicable. To ensure data compliance, a different analyst performs a second level of review. Second level review is accomplished by checking reported results against raw data and evaluating the results for accuracy. During the second level review, blank runs, QA/QC check results, continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. Manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:
  - QC data are outside the specified control limits for accuracy and precision
  - Reviewed sample data does not match with reported results
  - Unusual detection limit changes are observed
  - Samples having unusually high results
  - Samples exceeding a known regulatory limit

- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range
- **19.14.4.3** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Director/Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.
- **19.14.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.
- **19.14.4.5** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.
- **19.14.4.6** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. The accounting personnel also check the report for any clerical or invoicing errors. When complete, the report is sent out to the client.
- **19.14.4.7** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

#### 19.14.5 Manual Integrations

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration documentation procedures are referenced in the applicable analytical method SOP's in the Manual integration section.

**19.14.5.1** The analyst must adjust baseline or the area of a peak in some situations, for example when two compounds are not adequately resolved or when a peak shoulder needs to be separated from the peak of interest. The analyst must use professional judgment and common sense to determine when manual integrating is required.

Analysts are encouraged to ask for assistance from a senior analyst or manager when in doubt.

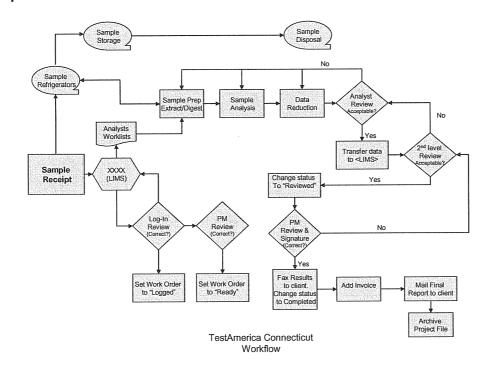
- **19.14.5.2** Analysts shall not increase or decrease peak areas to for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- **19.14.5.3** Client samples, performance evaluation samples, and quality control samples are all treated equally when determining whether or not a peak area or baseline should be manually adjusted.
- **19.14.5.4** All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

# Figure 19-1. Example - Demonstration of Capability Documentation

Demonstration of Capability Certification Statement				
Laboratory Name: Laboratory Address:	TestAmerica Conn 128 Long Hill Cros Shelton, CT 06484	s Road	Date:	
Method: Matrix:				
Analyst Name:				
*Analyst Name:				
We the undersigned	certify that:			
<ol> <li>The analyst identified above, using the cited test method, which is in use at this facility for the analysis of samples under the National Environmental Laboratory Accreditation Program, have met the Initial Demonstration of Capability.</li> <li>The test method was performed by the analyst identified on this certification.</li> <li>Copies of the test method and SOP are available for all personnel on site.</li> <li>The data associated with the DoC are true, complete and representative.</li> <li>All raw data (including a copy of this certification form) necessary to reconstruct and validate these analyses have been retained at the facility, and that the associated information is available for review by authorized inspectors.</li> </ol>				
Laboratory Manager	Supervisor	Signature		Date
Quality Assurance M	anager	Signature		Date

\* second analyst if sample prepped by another person

# Figure 19-2 Example: Work Flow



### SECTION 20

### EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

### 20.1 <u>OVERVIEW</u>

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in laboratory SOPs. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers instructions for equipment use are readily accessible to all appropriate laboratory personnel.

### 20.2 PREVENTIVE MAINTENANCE

**20.2.1** The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

**20.2.2** Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

**20.2.3** Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

**20.2.4** Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

**20.2.4.1** Documentation must include all major maintenance activities such as contracted preventive maintenance and service and in-house activities such as the replacement of electrical components, lamps, tubing, valves, columns, detectors, cleaning and adjustments.

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- **20.2.4.2** Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on *'date'* was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.
- **20.2.4.3** When maintenance or repair is performed by an outside agency, service receipts detailing the service performed are filed with the Department Manager.

**20.2.5** If an instrument requires repair (subjected to overloading or mishandling, gives suspect results, or otherwise has shown to be defective or outside of specified limits) it shall be taken out of operation and tagged as out-of-service or otherwise isolated until such a time as the repairs have been made and the instrument can be demonstrated as operational by calibration and/or verification or other test to demonstrate acceptable performance. The laboratory shall examine the effect of this defect on previous analyses.

**20.2.6** In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

**20.2.7** If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

# 20.3 SUPPORT EQUIPMENT

This section applies to all devices that may not be the actual test instrument, but are necessary to support laboratory operations. These include but are not limited to: balances, ovens, refrigerators, freezers, incubators, water baths, temperature measuring devices, and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

#### 20.3.1 Weights and Balances

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or

other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file. Refer to the SOP for Balance Calibration, CT-QAS-9.

#### 20.3.2 pH, Conductivity, and Turbidity Meters

The pH meters used in the laboratory are accurate to  $\pm$  0.1 pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

#### 20.3.3 <u>Thermometers</u>

All thermometers are calibrated on an annual basis with a NIST-traceable thermometer. IR thermometers, digital probes and thermocouples are calibrated quarterly.

The NIST thermometer is recalibrated every five years (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometer is used for no other purpose than to calibrate other thermometers.

All of this information is documented in logbooks. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logbooks. More information on this subject can be found in the SOP for Thermometer Calibration, CT-QAS-11.

## 20.3.4 <u>Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators</u>

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between > 0°C and  $\leq$  6 °C.

Specific temperature settings/ranges for other refrigerators, ovens waterbaths, and incubators can be found in method specific SOPs.

All of this information is documented in Daily Temperature Logbooks and method-specific logbooks.

## 20.3.5 Autopipettors, Dilutors, and Syringes

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis. Glass micro-syringes are considered the same as Class A glassware.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Receivers are filled to the meniscus with distilled water. The water is then decanted off to a calibrated weighing vessel. The weight is recorded and is converted to the proper volume. Calibration verfication procedures can be found in specific method SOPs that use this type of glassware.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. The laboratory keeps on file an "Accuracy and Precision Statement of Conformance" from Hamilton attesting established accuracy.

# 20.4 INSTRUMENT CALIBRATIONS

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response,

type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration.)

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:** Instruments are calibrated initially and as needed after that and at least annually

# 20.4.1 CALIBRATION STANDARDS

**20.4.1.1** Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP.

**20.4.1.2** Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

**20.4.1.3** The lowest concentration calibration standard that is analyzed during an initial calibration must be at or below the stated reporting limit for the method based on the final volume of extract (or sample).

**20.4.1.4** All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

### 20.4.2 <u>Calibration Verification</u>

The calibration relationship established during the initial calibration must be verified at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and NELAC (2003) standard, Section 5.5.5.10. The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models.

**Note:** The process of calibration verification referred to is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met.

All samples must be bracketed by periodic analyses of standards that meet the QC acceptance criteria (e.g., calibration and retention time). The frequency is found in the determinative methods or SOPs.

**Note:** If an internal standard calibration is being used (basically GCMS) then bracketing standards are not required, only daily verifications are needed. The results from these verification standards must meet the calibration verification criteria and the retention time criteria (if applicable).

# 20.4.2.1 Verification of Linear Calibrations

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs.) Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

# 20.4.2.2 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

# 20.5 TENTATIVELY IDENTIFIED COMPOUNDS (TICS) – GC/MS ANALYSIS

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

#### 20.6 GC/MS TUNING

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

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# Table 20-1. Example: Instrumentation List

Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
ICP	Thermo Jarrell Ash (61P) S/N 464790	61E Trace	1997	Yes	6010B, 200.7
ICP-MS	Agilent S/N JP51202170	7500 Series ICP- MS	2008	Yes	6020, 200.8
Mercury Anályzer	Perkin Elmer S/N 1398 509550	FIMS100	1999	Yes	7471A, 7470, 245.1
GC/MS Semivolatiles	Agilent (U) S/N US33210086	6890/5973	2004	Yes	8270C, 625, SIM
	Agilent (Z) S/N US52430633	6890/5975	2005	Yes	8270C, 625, SIM
	Agilent (A) S/N US52420834	6890/5975	2006	Yes	8270C, 625, SIM
	Agilent (C) S/N US52430481	6890/5975	2006	Yes	8270C, 625, SIM
GC/MS Volatiles	Agilent (L) S/N 3240A18492	5890/5971	1992	Yes	8260B, 624
	Agilent (O) S/N 3203A41807	5890/5971	1991	Yes	8260B, 624 – waters
	Agilent (N) S/N 3133A37851	5890/5971	1991	Yes	8260B, 624
	Agilent (W) S/N U544621422	6890/5973	2005	Yes	8260B, 624 – soils
	Agilent (Y) S/N U544621422	6890/5973	2005	Yes	8260B, 624
	Agilent (V) S/N U540620567	6890/5973	2004	Yes	8260B, 624
GC Semivolatiles	Agilent (GCX-C/D) S/N CN10832045	7890 - Dual FID	2008	Yes	CTETPH 8015B (DRO)
	Agilent (GC4C/D) S/N 3033A33529	5890II - Dual ECD	1992	Yes	8082
	Agilent (GC7C/D) S/N CN10416081	6890-Dual micro ECD	2004	Yes	8081, 8082, 608
	Agilent (GC8C/D) S/N CN10630046	6890-Dual micro ECD	2006	Yes	8081, 8082, 608
	Agilent (GC9C/D) S/N US00028263	6890-Dual micro ECD	2007	Yes	8081, 8082, 608

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Instrument Type	Manufacturer	Model	Purchase Date	Autosampler	Method Performed
	Agilent (GC2C/D) S/N 3033A32099	589011 – FID	1991	Yes	CTETPH 8015B (DRO)
	Agilent (GC3) S/N 3033A32563	5890 - FID	1991	Yes	8015B (DRO)
lon Chromatograph	Lachat S/N A83000-1476	Quickchem 8000	1999	Yes	300.0, 9056 350.1, 351.2 9012, 335.4, 353.2, 420.2
TOC	Dohrmann S/N 98315003	Phoenix 8000	2004	No	415.2, 9060
	Vario Elementar III S/N 11054049	Vario EL	2005	Yes	415.2, 9060, Lloyd Kahn
TKN Digestion System	Scientific Instruments	AD-4020	1994	No	351.2, 351.3
UV/VIS	Thermo electron	Genesys 10	2006	No	7196A, 365.1 or Equiv.
UV/VIS	Buck Scientific (not in use)	HC 404	2000	No	418.1
PH Meter	Orion Research (not in use)	SA 720	1998	No	9040B, 9045C, 150.1
PH Meter VWR		8025		No	9040B, 9045C, 150.1
Autotitrator (pH, Alkalinity, Conductance)	Man-Tech (ATZ) S/N MS-0A3-615	PC 1300-475	2003	Yes	9040B, 9045C, 150.1, 2320B, 310.1, 310.2, 2510B, 9050A, 120.1
Dissolved Oxygen Meter	YSI	51A	1994	No	405.1
Turbidimeter	НАСН	2100 N	1990	No	180.1
Conductivity	Cole-Parmer	1484-20	1996	No	120.1
Automated Distillation Apparatus	Westco S/N 1028	1075 Easy Dist	2003	No	350.1, 420.2, 9066
COD	НАСН	45600	1991	No	410.4
Flash Point Apparatus	ERDCO	RT-00001		No	1020
Midi Distillation Setups	Andrews Galss	110-10-R	1995	No	9012A, 335.1, 335.3
TCLP Spinners	Dayton	3M137B/5K939B	1990	No	1311, 1312
GPC			1999	Yes	8270, 8081, 8082
Selective Chemistry Analyzer	Thermo electron S/N E1519588	Konelab Aqua 20	2004	Yes	350.1, 351.2, 353.2, 365.2
Solvent Evaporator	Horizon Technology	Speed-Vap III	2004	No	1664A
Colorimeter	Hach	DR/890		No	410.4

# Table 20-2. Schedule of Routine Maintenance

Instrument	Procedure	Frequency
Leeman Mercury Analyzer	Check tubing for wear Replace lamps Insert clean drying tube filled with Magnesium Perchlorate Fill reductant bottle with 10% Stannous Chloride	Daily As required Daily Daily
ICP	Check pump tubing Check liquid argon supply Check fluid level in waste container Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Replace pump tubing	Daily Daily Daily Daily Monthly As required Monthly As required
ICP-MS	Check pump tubing Check liquid argon supply Check fluid level in waste container Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Replace pump tubing Clean/Replace Sample Cones/Skimmer Cones	Daily Daily Daily Daily Monthly As required Monthly As required As required
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Inspect line coils, heating baths and filters Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly
Agilent GC/MS	Clean injection ports Pump oil-level check Pump oil changing Ion source cleaning and filament replacement Replace electron multiplier Change exhaust trap absorbent Inspect and refill the calibration sample vial with PFTBA Vacuum fan grills and filters Change liners and septum Column replacement and conditioning Column cutting and reinstallation	As required Monthly Annually As required As required Every 6 months As required Every 6 months As required As required As required

Instrument	Procedure	Frequency
Gas Chromatograph	Injection port cleaning Septum replacement Check for loose/frayed wires and insulation Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	As required As required As Required As Required As Required As Required
Purge and Trap concentrators/ Archon	Check purge flow Inspect line and valve temperatures Change and condition trap Adjust purge flow Rinse sample lines Bake out trap Replace lines and fittings Check syringe Check reagent water and waste bottles Autocalibrate robotic arm Replace inline filter	Daily Daily As required As required As required After each analysis, extend as needed As required Daily Daily As required As required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Daily conductivity check System cleaning Replace cartridge & large mixed bed resins	Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/ Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

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### SECTION 21

### MEASUREMENT TRACEABILITY (NELAC 5.5.6)

### 21.1 <u>OVERVIEW</u>

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices. (Refer to Section 20.3). With the exception of Class A Glassware (including glass microliter syringes that have a certificate of accuracy), quarterly accuracy checks are performed for all mechanical volumetric devices. Microsyringes are verified at least semi-annually or disposed of after 6 months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A glassware should be routinely inspected for chips, acid etching or deformity. If the Class A glassware is suspect, the accuracy of the glassware will be assessed prior to use.

## 21.2 NIST-TRACEABLE WEIGHTS AND THERMOMETERS

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

An external certified service engineer services laboratory balances on an annual basis. This service is documented on each balance with a signed and dated certification sticker. Balance calibrations are checked each day of use. All mercury thermometers are calibrated annually against a traceable reference thermometer. Temperature readings of ovens, refrigerators, and incubators are checked on each day of use.

### 21.3 REFERENCE STANDARDS / MATERIALS

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number and expiration date. All documentation received with the reference standard is retained as a QC record and references the Standard Identification Number.

All reference, primary and working standards/materials, whether commercially purchased or laboratory prepared, must be checked regularly to ensure that the variability of the standard or material from the 'true' value does not exceed method requirements. The accuracy of calibration standards is checked by comparison with a standard from a second source. In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an Initial Calibration Verification (ICV) or LCS (where there is no sample preparation) is used as the second source confirmation. These checks are generally performed as an integral part of the analysis method (e.g. calibration checks, laboratory control samples).

All standards and materials must be stored and handled according to method or manufacturer's requirements in order to prevent contamination or deterioration. Refer to the Corporate Environmental Health & Safety Manual or laboratory SOPs. For safety requirements, please refer to method SOPs and the laboratory Environmental Health and Safety Manual.

# 21.4 <u>DOCUMENTATION AND LABELING OF STANDARDS, REAGENTS, AND</u> <u>REFERENCE MATERIALS</u>

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP (CA-Q-S-001), Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificate of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained within the departments. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on documentation and labeling, please refer to method specific SOPs.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc.., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

**21.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department
- Preparer's name

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- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date (include prep date for reagents)
- Standard ID (generated by the LIMS system)
- Special Health/Safety warnings if applicable

**21.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include a preparation date, expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

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All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

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# **SECTION 22**

## SAMPLING (NELAC 5.5.7)

## 22.1 <u>OVERVIEW</u>

The laboratory does not provide sampling services. The laboratory's responsibility in the sample collection process lies in supplying the sampler with the necessary coolers, reagent water, sample containers, preservatives, sample labels, custody seals, COC forms, ice, and packing materials required to properly preserve, pack, and ship samples to the laboratory

### 22.2 SAMPLING CONTAINERS

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory.

## 22.2.1 <u>Preservatives</u>

Upon request, preservatives are provided to the client in pre-cleaned sampling containers. In some cases containers may be purchased pre-preserved from the container supplier. Whether prepared by the laboratory or bought pre-preserved, the grades of the preservatives are at a minimum:

- Hydrochloric Acid Reagent ACS (Certified VOA Free) or equivalent
- Methanol Purge and Trap grade
- Nitric Acid Instra-Analyzed or equivalent
- Sodium Bisulfate ACS Grade or equivalent
- Sodium Hydroxide Instra-Analyzed or equivalent
- Sulfuric Acid Instra-Analyzed or equivalent
- Sodium Thiosulfate ACS Grade or equivalent

# 22.3 DEFINITION OF HOLDING TIME

The date and time of sampling documented on the COC form establishes the day and time zero. As a general rule, when the maximum allowable holding time is expressed in "days" (e.g., 14 days, 28 days), the holding time is based on calendar day measured. Holding times expressed in "hours" (e.g., 6 hours, 24 hours, etc.) are measured from date and time zero. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

### 22.4 SAMPLING CONTAINERS, PRESERVATION REQUIREMENTS, HOLDING TIMES

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The preservation and holding time criteria used by the laboratory are derived from the source documents for the methods and are listed in the lab's SOPs. If method required holding times this info is in the SOPs or preservation requirements are not met, the reports will be qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

# 22.5 SAMPLE ALIQUOTS / SUBSAMPLING

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative subsample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located in the SOP for Compositing, Homogenization and Subsampling Environmental Samples, # CT-SMS-11.

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## **SECTION 23**

### HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal.

# 23.1 CHAIN OF CUSTODY (COC)

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

## 23.1.1 Field Documentation

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name.

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The samples are stored in a cooler with ice, as applicable, and remain solely in the possession of the client's field technician until the samples are delivered to the laboratory. The sample collector must assure that each container is in his/her physical possession or in his/her view at all times, or stored in such a place and manner to preclude tampering. The field technician relinquishes the samples in writing on the COC form to the sample control personnel at the laboratory or to a TestAmerica courier. Samples are only considered to be received by lab when personnel at the laboratory have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. Airbills from the courier are stored in the log-infolder. Tracking numbers are entered into the LIMS in the cooler receipt comments field.

# 23.1.2 Legal / Evidentiary Chain-of-Custody

If samples are identified for legal/evidentiary purposes on the COC, login will complete the custody seal retain the shipping record with the COC, and initiate an internal COC for laboratory use by analysts and a sample disposal record.

# 23.2 SAMPLE RECEIPT

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections.

# 23.2.1 Laboratory Receipt

When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented in the LIMS sample Receipt check list and brought to the immediate attention of the Project Manager and subsequently the Client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

# 23.2.1.1 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- a COC filled out completely;
- samples must be properly labeled;
- proper sample containers with adequate volume for the analysis (Sampling Guide) and necessary QC;

- samples must be preserved according to the requirements of the requested analytical method (Sampling Guide);
- sample holding times must be adhered to (Sampling Guide);
- all samples submitted for water/solid Volatile Organic analyses must have a Trip Blank submitted at the same time;
- Apparent tampering of cooler and/or samples
- Temperature specifications not met
- the project manager will be notified if any sample is received in damaged condition.

Data from samples which do not meet these criteria are flagged and the nature of the variation from policy is defined. A copy of the sample acceptance policy is provided to each client prior to shipment of samples.

- **23.2.1.2** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.
- **23.2.1.3** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:
  - Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
  - Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according to the SOP for Sample Processing at Sample arrival, SOP No. CT-SMS-4.

# 23.3 <u>SAMPLE STORAGE</u>

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators suitable for the sample matrix. In addition, samples to be analyzed for volatile organic parameters are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All unused portions of samples, including empty sample containers(for soils), are returned to the secure sample control area. Empty water containeres are disposed of by the department using the sample.

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All samples are kept in the refrigerators for 30 days after invoice prior to disposal. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked at all times unless a project specifically demands it. Samples are accessible to laboratory personnel only. Visitors to the laboratory are prohibited from entering the refrigerator and laboratory areas unless accompanied by an employee of TestAmerica.

## 23.4 HAZARDOUS SAMPLES AND FOREIGN SOILS

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in an isolated area designated for hazardous waste only. Samples should be received into the lab as outlined in the SOPs for sample receiving.

If samples are received with additional paperwork indicating samples are from foreign sources then the samples must be handled and disposed of accordingly. Foreign source samples must be identified as needing special handling upon disposal by placing a green sticker on the top of the jar lid. Mixed waste radiological samples are identified with an orange sticker.

Foreign soil samples are autoclaved then sent out for incineration by a USDA-approved waste hauler.

### 23.5 SAMPLE SHIPPING

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses. The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

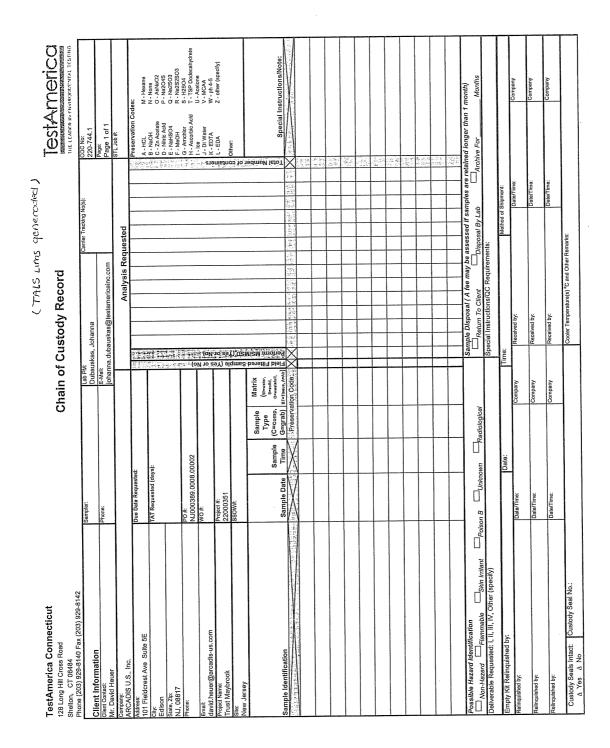
### 23.6 SAMPLE DISPOSAL

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP for Sample Disposal: #CT-SMS-14. All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory

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no longer than three months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according to state or federal guidelines may be returned to the client upon completion of the analytical work.

If a sample is part of a known litigation, the affected legal authority, sample data user, and/or submitter of the sample must participate in the decision about the sample's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file. Pertinent information includes the date of disposal, nature of disposal (such as sample depletion, hazardous waste facility disposal, return to client), names of individuals who conducted the arrangements and physically completed the task. The laboratory will remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated). A Waste Disposal Record should be completed.



# Example: Chain of Custody (COC)

Figure 23-1.

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## Figure 23-2

### Example: Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

- 1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
  - > Client name, address, phone number and fax number (if available)
  - > Project name and/or number
  - > The sample identification
  - > Date, time and location of sampling
  - > The collectors name
  - > The matrix description
  - > The container description
  - > The total number of each type of container
  - > Preservatives used
  - > Analysis requested
  - > Requested turnaround time (TAT)
  - > Any special instructions
  - > Purchase Order number or billing information (e.g. quote number) if available
  - > The date and time that each person received or relinquished the sample(s), including their signed name.
  - > The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab, and they must be exactly the same.
  - > Information must be legible
- 2) Samples must be properly labeled.
  - > Use durable labels (labels provided by TestAmerica are preferred)
  - > Include a unique identification number
  - > Include sampling date and time & sampler ID
  - > Include preservative used.
  - > Use indelible ink
  - > Information must be legible
- 3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested. See Lab Sampling Guide.
- 4) Samples must be preserved according to the requirements of the requested analytical method (See Sampling Guide.
- 5) Most analytical methods require chilling samples to 4° C (other than water samples for metals analysis). For these methods, the criteria are met if the samples are chilled to below 6° C. **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time

to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified upon receipt with the exception of volatiles and the project manager will be notified immediately if there is a discrepancy. Improperly preserved samples will be adjusted and recorded through a preservative sheet and NCM.
- 6) Sample Holding Times
  - TestAmerica will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48hr HT) sample must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.
  - Analyses that are designated as "field" analyses (Odor, pH, Dissolved Oxygen, Disinfectant Residual; a.k.a. Residual Chlorine, and Redox Potential) should be analyzed ASAP by the field sampler prior to delivering to the lab (within 15 minutes). However, if the analyses are to be performed in the laboratory, TestAmerica will make every effort to analyze the samples within 24 hours from receipt of the samples in the testing laboratory. Samples for "field" analyses received after 4:00 pm on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday unless a holiday). Samples will remain refrigerated and sealed until the time of analysis. Samples analyzed in the laboratory will be qualified on the final report with an 'H' to indicate holding time exceedance.
- 7) The project manager will be notified if any sample is received in damaged condition. TestAmerica will request that a sample be resubmitted for analysis.
- 8) Recommendations for packing samples for shipment.
  - > Pack samples in Ice rather than "Blue" ice packs.
  - > Soil samples should be placed in bubble wrap or bubble bags to help prevent breakage upon shipment. TestAmerica will supply these bags with the bottle order.
  - > Water samples would be best if wrapped with bubble-wrap or bubble bags to help prevent breakage upn shipment. TestAmerica will supply these bags with the bottle order.
  - ➢ Fill extra cooler space with bubble wrap.

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# Figure 23-3.

# Example: Cooler Receipt Form

TestAr	TestAmerica - Connecticut	ticut							
Trip Blank:									
QC:	Air:					Date Received:			
FB:						Sample #s:			
Soil:	Water:	H				Locations:			I
Laboratory Sample #	Relinquished by	Accepted by	Date	Time	Reason	Relinquished by	Accepted by	Date	Time
TestAmerica Fo	TestAmerica Form# SMF00508.CT								

## **SECTION 24**

## ASSURING THE QUALITY OF TEST RESULTS (NELAC 5.5.9)

## 24.1 <u>OVERVIEW</u>

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control measurements (e.g. Blanks, Laboratory Control Samples (LCS), Matrix Spikes (MS), duplicates (DUP), surrogates, Internal Standards (IS)). These quality control checks are performed as required by the method or regulations to assess precision and accuracy. In addition to the routine process quality control samples, Proficiency Testing (PT) Samples (concentrations unknown to laboratory) are analyzed to help ensure laboratory performance.

# 24.2 <u>CONTROLS</u>

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, grinding, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, drying and ashing. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

# 24.3 NEGATIVE CONTROLS

Control Type	Details
Method Blank (MB)	are used to assess preparation and analysis for possible contamination during the preparation and processing steps.
	The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
	The method blank is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (e.g., Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples.
	The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
Calibration Blanks	are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.

Table 24-1. Example – Negative Controls

Control Type	Details
Trip Blank <sup>1</sup>	are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples.
Field Blanks <sup>1</sup>	are sometimes used for specific projects by the field samplers. A field blank prepared in the field by filling a clean container with pure reagent water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)
Equipment Blanks <sup>1</sup>	are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)
Holding Blanks	also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

# 24.4 <u>POSITIVE CONTROLS</u>

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) or Sample Duplicate (MD, DUP), which evaluates field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

### 24.4.1 Method Performance Control - Laboratory Control Sample (LCS)

- **24.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.
- **24.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is

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made of a material containing known and verified amounts of analytes, taken through all preparation and analysis steps along with the field samples. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), a calibration verification standard is reported as the LCS. In some instances where there is no practical clean solid matrix available, aqueous LCS's may be processed for solid matrices; final results may be calculated as mg/kg or ug/kg, assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

- **24.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).
- **24.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples.
- 24.4.1.5 If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components of each spiking mix shall represent all chemistries, elution patterns and masses, permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.
  - **24.4.1.5.1** For methods that have 1-10 target analytes, spike all components.
  - **24.4.1.5.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
  - 24.4.1.5.3 For methods with more than 20 target analytes, spike at least 16 components.
  - **24.4.1.5.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
  - **24.4.1.5.5** Exception: Due to analyte incompatibility between the various PCB aroclors, aroclors 1016 and 1260 are used for spiking as they cover the range of all of the aroclors. Specific aroclors may be used by request on a project specific basis.

# 24.5 SAMPLE MATRIX CONTROLS

#### Table 24-2. Sample Matrix Control

Control Type		Details
Matrix Spikes (MS)	Use	used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used;
	Typical Frequency <sup>1</sup>	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency <sup>1</sup>	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSD's are normally not performed except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

# 24.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)

**24.6.1** As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

**24.6.2** Once control limits have been established, they are verified, reviewed, and updated if necessary on an annual basis unless the method requires more frequent updating. Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**24.6.3** Laboratory generated % Recovery acceptance (control) limits are generally established by taking  $\pm$  3 Standard Deviations (99% confidence level) from the average recovery of a minimum of 20-30 data points (more points are preferred).

- **24.6.3.1** Regardless of the calculated limit, the limit should be no tighter than the Calibration Verification (ICV/CCV). (Unless the analytical method specifies a tighter limit).
- 24.6.3.2 In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- **24.6.3.3** The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- **24.6.3.4** The maximum acceptable recovery limit will be 150%.
- **24.6.3.5** The maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 10%.
- **24.6.3.6** If either the high or low end of the control limit changes by  $\leq 5\%$  from previous, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.4** The lab must be able to generate a current listing of their control limits and track when the updates are performed. In addition, the laboratory must be able to recreate historical control limits. Refer to the SOP for Control Charts, QAS02601.ct.

24.6.4.1 One example: The QA department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses performed at TestAmerica Connecticut. This summary includes an effective date, is updated each time new limits are generated and is located with the QA manager. Unless otherwise noted, limits within these tables are laboratory generated. The analysts are instructed to use the current limits in the laboratory (dated and approved by the Technical Director and QA Manager) and entered into

the Laboratory Information Management System (LIMS). The Quality Assurance department maintains an archive of all limits used within the laboratory.

**24.6.5** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- **24.6.5.1** The analyte results are below the reporting limit and the LCS is above the upper control limit.
- **24.6.5.2** If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

**24.6.6** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.7** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

# 24.7 ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL

**24.7.1** The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

**24.7.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

24.7.3 Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

- **24.7.4** Selection of appropriate reagents and standards is included in Section 9 and 21.
- **24.7.5** A discussion on selectivity of the test is included in Section 5.

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- **24.7.6** Constant and consistent test conditions are discussed in Section 18.
- **24.7.7** The laboratories sample acceptance policy is included in Section 23.

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### **SECTION 25**

#### **REPORTING RESULTS (NELAC 5.5.10)**

#### 25.1 <u>OVERVIEW</u>

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

### 25.2 <u>TEST REPORTS</u>

Analytical results are reported in a format that is satisfactory to the client and meets all requirements of applicable accrediting authorities and agencies. A variety of report formats are available to meet specific needs. The report is printed, reviewed, and signed by the appropriate signatory. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1** A report title (e.g. Analytical Report For Samples) with a "sample results" column header.

**25.2.2** Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.

**25.2.3** A unique identification of the report (e.g. Job number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:** Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4** A copy of the chain of custody (COC).

• Any COCs involved with Subcontracting are included.

 Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (eg. Sampling information).

**25.2.5** The name and address of client and a project name/number, if applicable.

25.2.6 Client project manager or other contact

**25.2.7** Description and unambiguous identification of the tested sample(s) including the client identification code.

**25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.

**25.2.9** Date reported or date of revision, if applicable.

**25.2.10** Method of analysis including method code (EPA, Standard Methods, etc).

**25.2.11** Practical quantitation limits or reporting limit.

**25.2.12** Method detection limits (if requested)

**25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).

**25.2.14** Sample results.

**25.2.15** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.

**25.2.16** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 3 regarding additional addenda).

**25.2.17** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.

**25.2.18** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory coordinator.

**25.2.19** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.

**25.2.20** When NELAC accreditation is required, the lab shall certify that the test results meet all requirements of NELAC or provide reasons and/or justification if they do not.

**25.2.21** The laboratory includes a cover letter.

**25.2.22** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.23** When soil samples are analyzed, a specific identification as to whether soils are reported on a "wet weight" or "dry weight" basis.

**25.2.24** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.25** If only part of the report is provided to the client (client requests some results before all of it is complete), it must be clearly indicated on the report (e.g., partial report). A complete report must be sent once all of the work has been completed.

**25.2.26** Any non-TestAmerica subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All TestAmerica subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

Note: Refer to the Corporate SOP on Electronic Reporting and Signature Policy (No. CA-I-P-002) for details on internally applying electronic signatures of approval.

# 25.2.27 REPORTING LEVEL OR REPORT TYPE

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I is a report with the features described in Section 25.2 above.
- Level II is a Level I report plus summary information, including results for the method blank reported to the laboratory MDL, percent recovery for laboratory control samples and matrix spike samples, and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level II, but presented on the CLP-like summary forms, and relevant calibration information. A Level II report is not included, unless specifically requested. No raw data is provided.
- Level IV is the same as Level III with the addition of all raw supporting data.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.7.

# 25.2.28 Electronic Data Deliverables (EDDs)

EDDs are routinely offered as part of TestAmerica's services. TestAmerica Connecticut offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), NJ Haz Site, Standard Excel, Dbase, GISKEY, and EQuis.

EDD specifications are submitted to the IT department by the Data Reporting Department for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

# 25.3 SUPPLEMENTAL INFORMATION FOR TEST

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

**25.3.1** Numeric results with values outside of the calibration range, either high or low are gualified as 'estimated'.

**25.3.2** Where quality system requirements are not met, a statement of compliance/noncompliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet NELAC sample acceptance requirements such as improper container, holding time, or temperature.

**25.3.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**25.3.4** Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

# 25.4 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP # CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

# 25.5 CLIENT CONFIDENTIALITY

In situations involving the transmission of environmental test results by telephone, facsimile or other electronic means, client confidentiality must be maintained.

TestAmerica will not intentionally divulge to any person (other than the Client or any other person designated by the Client in writing) any information regarding the services provided by TestAmerica or any information disclosed to TestAmerica by the Client. Furthermore, information known to be potentially endangering to national security or an entity's proprietary rights will not be released.

**Note:** This shall not apply to the extent that the information is required to be disclosed by TestAmerica under the compulsion of legal process. TestAmerica will, to the extent feasible, provide reasonable notice to the client before disclosing the information.

**Note:** Authorized representatives of an accrediting authority are permitted to make copies of any analyses or records relevant to the accreditation process, and copies may be removed from the laboratory for purposes of assessment.

**25.5.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

This material is intended only for the use of the individual(s) or entity to whom it is addressed, and may contain information that is privileged and confidential. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this communication is strictly prohibited. If you have received this communication in error, please notify us immediately by telephone at the 1-203-929-8140 (or for e-mails: please notify us immediately by e-mail or by phone (1-203-929-8140) and delete this material from any computer).

# 25.6 FORMAT OF REPORTS

The format of reports is designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

# 25.7 <u>AMENDMENTS TO TEST REPORTS</u>

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the LIMS server, as is the original report. The revised report is stored under the Job Deliverables and identified with a revision number.

When the report is re-issued, a notation of "Revision #" is placed on the cover/signature page of the report *or at the top of the narrative page* with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example: Report was revised on 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.* 

# 25.8 POLICIES ON CLIENT REQUESTS FOR AMENDMENTS

## 25.8.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

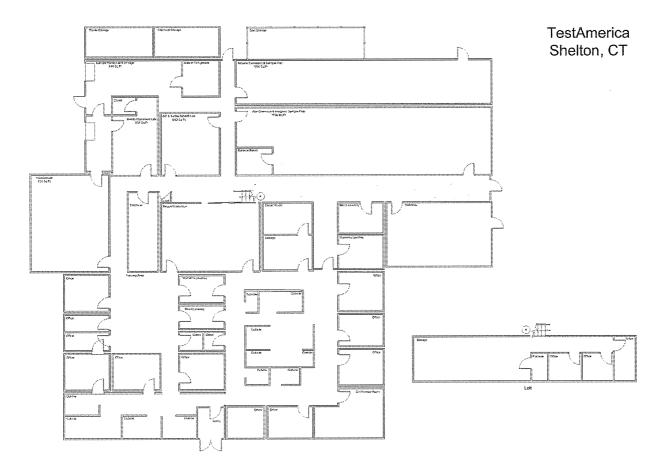
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely <u>no possible</u> impact on the interpretation of the analytical results and there is <u>no possibility</u> of the change being interpreted as misrepresentation by anyone inside or outside of our company.

### 25.8.2 <u>Multiple Reports</u>

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

# Appendix 1.

# Laboratory Floor Plan



### Appendix 2. Glossary/Acronyms

#### Glossary:

#### Acceptance Criteria:

Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

#### Accreditation:

The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)

#### Accrediting Authority:

The Territorial, State, or Federal Agency having responsibility and accountability for environmental laboratory accreditation and which grants accreditation (NELAC) [1.5.2.3]

#### Accuracy:

The degree of agreement between an observed value and an accepted reference value. Accuracy includes a combination of random error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS)

#### Analyst:

The designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)

#### Batch:

Environmental samples which are prepared and/or analyzed together with the same process and personnel, using the same lot(s) of reagents. A preparation batch is composed of one to 20 environmental samples of the same matrix, meeting the above mentioned criteria and with a maximum time between the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples (extracts, digestates or concentrates) and /or those samples not requiring preparation, which are analyzed together as a group using the same calibration curve or factor. An analytical batch can include samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Committee)

#### <u>Blank:</u>

A sample that has not been exposed to the analyzed sample stream in order to monitor contamination during sampling, transport, storage or analysis. The blank is subjected to the usual analytical and measurement process to establish a zero baseline or background value and is sometimes used to adjust or correct routine analytical results. (ASQC)

#### Blind Sample:

A sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst's or laboratory's proficiency in the execution of the measurement process.

#### Calibration:

To determine, by measurement or comparison with a standard, the correct value of each scale reading on a meter, instrument, or other device. The levels of the applied calibration standard should bracket the range of planned or expected sample measurements. (NELAC)

#### Calibration Curve:

The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

#### Calibration Method:

A defined technical procedure for performing a calibration. (NELAC)

#### Calibration Standard:

A substance or reference material used to calibrate an instrument (QAMS)

#### Certified Reference Material (CRM):

A reference material one or more of whose property values are certified by a technically valid procedure, accompanied by or traceable to a certificate or other documentation which is issued by a certifying body. (ISO Guide 30–2.2)

#### Chain of Custody:

An unbroken trail of accountability that ensures the physical security of samples and includes the signatures of all who handle the samples. (NELAC) [5.12.4]

#### Clean Air Act:

The enabling legislation in 42 U>S>C> 7401 et seq., Public Law 91-604, 84 Stat. 1676 Pub. L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and enforce them. (NELAC)

<u>Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/SUPERFUND):</u> The enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)

#### **Compromised Samples:**

Those samples which are improperly sampled, insufficiently documented (chain of custody and other sample records and/or labels), improperly preserved, collected in improper containers, or exceeding holding times when delivered to a laboratory. Under normal conditions, compromised samples are not analyzed. If emergency situation require analysis, the results must be appropriately qualified. (NELAC)

#### Confidential Business Information (CBI):

Information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information identified as such in full confidentiality.

#### **Confirmation:**

Verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:

Second column confirmation Alternate wavelength Derivatization Mass spectral interpretation Alternative detectors or Additional Cleanup procedures

#### (NELAC)

#### Conformance:

An affirmative indication or judgement that a product or service has met the requirements of the relevant specifications, contract, or regulation; also the state of meeting the requirements. (ANSI/ASQC E4-1994)

<u>Correction</u>: Actions necessary to correct or repair analysis specific non-conformances. The acceptance criteria for method specific QC and protocols as well as the associated corrective actions. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

### **Corrective Action:**

The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

#### Data Audit:

A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria). (NELAC)

#### Data Reduction:

The process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

#### Deficiency:

An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

#### **Detection Limit:**

The lowest concentration or amount of the target analyte that can be identified, measured, and reported with confidence that the analyte concentration is not a false positive value. See Method Detection Limit. (NELAC)

### Document Control:

The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

#### Duplicate Analyses:

The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

#### Environmental Detection Limit (EDL):

The smallest level at which a radionuclide in an environmental medium can be unambiguously distinguished for a given confidence interval using a particular combination of sampling and measurement procedures, sample size, analytical detection limit, and processing procedure. The EDL shall be specified for the 0.95 or greater confidence interval. The EDL shall be established initially and verified annually for each test method and sample matrix. (NELAC Radioanalysis Subcommittee)

#### Equipment Blank:

Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (NELAC)

#### External Standard Calibration:

Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

### Federal Water Pollution Control Act (Clean Water Act, CWA):

The enabling legislation under 33 U.S.C. 1251 et seq., Public Law 92-50086 Stat 816, that empowers EPA to set discharge limitations, write discharge permits, monitor, and bring enforcement action for non-compliance. (NELAC)

#### Field Blank:

Blank prepared in the field by filing a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken (EPA OSWER)

#### Field of Testing:

NELAC's approach to accrediting laboratories by program, method and analyte. Laboratories requesting accreditation for a program-method-analyte combination or for an up-dated/improved method are required to submit to only that portion of the accreditation process not previously addressed (see NELAC, section 1.9ff). (NELAC)

#### Holding Times (Maximum Allowable Holding Times):

The maximum times that samples may be held prior to analyses and still be considered valid or not compromised. (40 CFR Part 136)

#### Internal Standard:

A known amount of standard added to a test portion of a sample and carried through the entire measurement process as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (NELAC)

#### Internal Standard Calibration:

Calibrations for methods that utilize internal standards to compensate for changes in instrument conditions.

#### Instrument Blank:

A clean sample (e.g., distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

Laboratory Control Sample (however named, such as laboratory fortified blank, spiked blank, or QC check sample):

A sample matrix, free from the analytes of interest, spiked with verified known amounts of analytes or a material containing known and verified amounts of analytes, taken through all preparation and analysis steps. Where there is no preparation taken for an analysis (such as in aqueous volatiles), or when all samples and standards undergo the same preparation and analysis process (such as Phosphorus), there is no LCS. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system.

An LCS shall be prepared at a minimum of 1 per batch of 20 or less samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The results of these samples shall be used to determine batch acceptance.

Note: NELAC standards allow a matrix spike to be used in place of this control as long as the acceptance criteria are as stringent as for the LCS. (NELAC)

#### Laboratory Duplicate:

Aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)

### Least Squares Regression (1<sup>st</sup> Order Curve):

The least squares regression is a mathematical calculation of a straight line over two axes. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The regression calculation will generate a correlation coefficient (r) that is a measure of the "goodness of fit" of the regression line to the data. A value of 1.00 indicates a perfect fit.

In order to be used for quantitative purposes, r must be greater than or equal to 0.99 for organics and 0.995 for inorganics.

#### Limit of Detection (LOD):

An estimate of the minimum amount of a substance that an analytical process can reliably detect. An LOD is analyte- and matrix-specific and may be laboratory dependent. (Analytical Chemistry, 55, p.2217, December 1983, modified) See also Method Detection Limit.

#### <u>Matrix:</u>

The component or substrate that contains the analyte of interest. For purposes of batch and QC requirement determinations, the following matrix distinctions shall be used:

Aqueous: Any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

Drinking Water: any aqueous sample that has been designated as a potable or potential potable water source.

Saline/Estuarine: any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.

Non-aqueous Liquid: any organic liquid with <15% settleable solids.

Biological Tissue: any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.

Solids: includes soils, sediments, sludges, and other matrices with >15% settleable solids.

Chemical Waste: a product or by-product of an industrial process that results in a matrix not previously defined.

Air: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbant tube, impinger solution, filter, or other device. (NELAC)

#### Matrix Spike (spiked sample or fortified sample):

Prepared by adding a known mass of target analyte to a specified amount of matrix sample for which an independent estimate of target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the matrix on a method's recovery efficiency.

Matrix spikes shall be performed at a frequency of one in 20 samples per matrix type per sample extraction or preparation method except for analytes for which spiking solutions are not available such as, total suspended solids, total dissolved solids, total volatile solids, total solids, pH, color, odor, temperature, dissolved oxygen or turbidity. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in a matrix spike may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the spike. (QAMS)

#### Matrix Spike Duplicate (spiked sample or fortified sample duplicate):

A second replicate matrix spike is prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte.

Matrix spike duplicates or laboratory duplicates shall be analyzed at a minimum of 1 in 20 samples per matrix type per sample extraction or preparation method. The laboratory shall document their procedure

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to select the use of an appropriate type of duplicate. The selected sample(s) shall be rotated among client samples so that various matrix problems may be noted and/or addressed. Poor performance in the duplicates may indicate a problem with the sample composition and shall be reported to the client whose sample was used for the duplicate. (QAMS)

#### Method Blank:

A sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses. (NELAC)

#### Method Detection Limit:

The minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte. (40 CFR Part 136, Appendix B)

#### Negative Control:

Measures taken to ensure that a test, its components, or the environment do not cause undesired effects, or produce incorrect test results. (NELAC)

#### Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

#### Performance Based Measurement System (PBMS):

A set of processes wherein the data quality needs, mandates or limitations of a program or project are specified and serve as criteria for selecting appropriate test methods to meet those needs in a cost-effective manner. (NELAC)

#### Positive Control:

Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)

#### Precision:

The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator. Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC)

#### Preservation:

Refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)

#### Proficiency Testing:

A means of evaluating a laboratory's performance under controlled conditions relative to a given set of criteria through analysis of unknown samples provided by an external source. (NELAC) [2.1]

#### Proficiency Testing Program:

The aggregate of providing rigorously controlled and standardized environmental samples to a laboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories. (NELAC)

#### Proficiency Test Sample (PT):

A sample, the composition of which is unknown to the analyst and is provided to test whether the analyst/laboratory can produce analytical results within specified acceptance criteria. (QAMS)

#### Quality Assurance:

An integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS)

### Quality Assurance [Project] Plan (QAPP):

A formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EAP-QAD)

### Quality Control:

The overall system of technical activities which purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS)

### Quality Control Sample:

An uncontaminated sample matrix spiked with known amounts of analytes from a source independent from the calibration standards. It is generally used to establish intra-laboratory or analyst specific precision and bias or to assess the performance of all or a portion of the measurement system. (EPA-QAD)

### Quality Manual:

A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, to ensure the quality of its product and the utility of its product to its users. (NELAC)

#### Quality System:

A structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring quality in its work processes, products (items), and services. The quality system provides the framework for planning, implementing, and assessing work performed by the organization and for carrying out required QA and QC (ANSI/ASQC-E-41994)

### **Quantitation Limits:**

The maximum or minimum levels, concentrations, or quantities of a target variable (e.g., target analyte) that can be quantified with the confidence level required by the data user. (NELAC)

#### Range:

The difference between the minimum and the maximum of a set of values. (EPA-QAD)

### Reagent Blank (method reagent blank):

A sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

#### Reference Material:

A material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

### Reference Standard:

A standard, generally of the highest metrological quality available at a given location, from which measurements made at that location are derived. (VIM-6.0-8)

### Replicate Analyses:

The measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)

#### Report Limit (RL):

The laboratory nominal Quantitation Limit (QL) or the level of sensitivity required by the client but not lower than the LOD.

#### Resource Conservation and Recovery Act (RCRA):

The enabling legislation under 42 USC 321 et seq. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage, and disposal. (NELAC)

#### Safe Drinking Water Act (SDWA):

The enabling legislation, 42 USC 300f et seq. (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations. (NELAC)

#### Sample Duplicate:

Two samples taken from and representative of the same population and carried through all steps of the sampling and analytical procedures in an identical manner. Duplicate samples are used to assess variance of the total method including sampling and analysis. (EPA-QAD)

<u>Second Order Polynomial Curve (Quadratic)</u>: The 2<sup>nd</sup> order curves are a mathematical calculation of a slightly curved line over two axis. The y axis represents the instrument response (or Response ratio) of a standard or sample and the x axis represents the concentration. The 2<sup>nd</sup> order regression will generate a coefficient of determination (COD or  $r^2$ ) that is a measure of the "goodness of fit" of the quadratic curvature the data. A value of 1.00 indicates a perfect fit. In order to be used for quantitative purposes,  $r^2$  must be greater than or equal to 0.99.

#### Selectivity:

(Analytical chemistry) the capability of a test method or instrument to respond to a target substance of constituent in the presence of non-target substances. (EPA-QAD)

### Sensitivity:

The capability of a method or instrument to discriminate between measurement responses representing different levels (e.g., concentrations) of a variable of interest. (NELAC)

#### Spike:

A known mass of target analyte added to a blank, sample or sub-sample; used to determine recovery efficiency or for other quality control purposes.

If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. However, in cases where the components interfere with accurate assessment (such as simultaneously spiking chlordane, toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, a representative number (at a minimum 10%) of the listed components may be used to control the test method. The selected components of each spiking mix shall represent all chemistries, elution patterns and masses permit specified analytes and other client requested components. However, the laboratory shall ensure that all reported components are used in the spike mixture within a two-year time period.. (NELAC)

### Standard:

The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)

#### Standard Operating Procedures (SOPs):

A written document which details the method of an operation, analysis, or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)

#### Standardized Reference Material (SRM):

A certified reference material produced by the U.S. National Institute of Standards and Technology or other equivalent organization and characterized for absolute content, independent of analytical method. (EPA-QAD)

#### Surrogate:

A substance with properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes.

Surrogate compounds must be added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. Poor surrogate recovery may indicate a problem with sample composition and shall be reported to the client whose sample produced poor recovery. (QAMS)

#### Systems Audit (also Technical Systems Audit):

A thorough, systematic, qualitative on-site assessment of the facilities, equipment, personnel, training, procedures, record keeping, data validation, data management, and reporting aspects of a total measurement system. (EPA-QAD)

#### Toxic Substances Control Act (TSCA):

The enabling legislation in 15 USC 2601 et seq., (1976) that provides for testing, regulating, and screening all chemicals produced or imported into the United States for possible toxic effects prior to commercial manufacture. (NELAC)

#### Traceability:

The property of a result of a measurement whereby it can be related to appropriate standards, generally international or national standards, through an unbroken chain of comparisons. (VIM-6.12)

#### Uncertainty:

A parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measured value.

### Acronyms:

BS – Blank Spike

- BSD Blank Spike Duplicate
- CAR Corrective Action Report
- CCV Continuing Calibration Verification
- CF Calibration Factor
- CFR Code of Federal Regulations
- COC Chain of Custody
- CRS Change Request Form
- DOC Demonstration of Capability
- DQO Data Quality Objectives
- DU Duplicate
- **DUP** Duplicate
- EHS Environment, Health and Safety
- EPA Environmental Protection Agency

GC - Gas Chromatography

GC/MS - Gas Chromatography/Mass Spectrometry

HPLC - High Performance Liquid Chromatography

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ICP - Inductively Coupled Plasma Atomic Emission Spectroscopy ICV - Initial Calibration Verification IDL - Instrument Detection Limit IH - Industrial Hygiene IS – Internal Standard LCS - Laboratory Control Sample LCSD - Laboratory Control Sample Duplicate LIMS – Laboratory Information Management System MDL – Method Detection Limit MS – Matrix Spike MSD - Matrix Spike Duplicate MSDS - Material Safety Data Sheet NELAC - National Environmental Laboratory Accreditation Conference NELAP - National Environmental Laboratory Accreditation Program PT – Performance Testing QAM - Quality Assurance Manual QA/QC - Quality Assurance / Quality Control QAPP - Quality Assurance Project Plan RF - Response Factor **RPD** – Relative Percent Difference RSD - Relative Standard Deviation SD - Standard Deviation SOP: Standard Operating Procedure TAT – Turn-Around-Time VOA - Volatiles VOC - Volatile Organic Compound

## Appendix 3.

### Laboratory Certifications, Accreditations, Validations

**TestAmerica Connecticut** maintains certifications, accreditations, certifications, and validations with numerous state and national entities. Programs vary but may include on-site audits, reciprocal agreements with another entity, performance testing evaluations, review of the QA Manual, Standard Operating Procedures, Method Detection Limits, training records, etc. At the time of this QA Manual revision, the laboratory has accreditation/certification/licensing with the following organizations:

State	Responsible Agency	Certification	Lab Number
Connecticut	Department of Health Services	Drinking Water, Wastewater	PH-0497
Maine	Department of Health and Environmental Services	Wastewater/Solid, Hazardous Waste	CT023
Massachusetts	Department of Environmental Protection	Potable/Non-Potable Water	CT023
New Hampshire	Department of Environmental Services	Drinking Water, Wastewater NELAC	2528
New Jersey	Department of Environmental Protection	Drinking Water, Wastewater <b>NELAC</b> *	CT410
New York	Department of Health	CLP, Drinking Water, Wastewater, Solid/ Hazardous Waste	10602
		NELAC	
Rhode Island	Department of Health	ChemistryNon- Potable Water and Wastewater	A43
Utah	Department of Health	RCRA-NELAC	2032614458

The certificates and parameter lists (which may differ) for each organization may be found on the corporate web site, the laboratory's public server, the final report review table, and in the following offices: QA, marketing, and project management.

\* Primary Accrediting Authority - NELAC

# Appendix 4.

# Laboratory Test Methods

Analysis	Method	Analysis	Method
GC/MS Volatiles Prep	5030B	COD	410.4
GC/MS Volatiles Prep	5035H	Total Residual Chlorine	SM4500CL G
GC/MS Volatiles Prep	5035L	Hexavalent Chromium	7196A
GC/MS Volatiles Prep	3585	Trivalent Chromium	SM3500 CR D
GC/MS Volatiles-TCLP	1311ZHE	Color	SM2120B
GC/MS Volatiles	624	Specific Conductance	120.1
GC/MS Volatiles	8260B	Specific Conductance	SM2510B
GC/MS Volatiles	OLM3.2	Cyanide (Amenable)	SM4500 CN G
GC/MS Volatiles	OLM4.3	Cyanide (Total)	335.4
GC/MS Volatiles	524.2	Cyanide (Total & Amenable)	9012B
GC/Svoa Prep - Waters	3510C	Free Cyanide	D4282 02
GC/Svoa Prep - Soils	3550B	CLP Cyanide	ILM4.0
GC/Svoa Prep - Soils	3541	Cyanide Prep – oils/soils	9013
GC/Svoa Prep - Oils	3580A	Ignitability	1030
Florisil Cleanup	3620B	Flashpoint	1020A
Sulfur Cleanup	3660B	Odor	140.1
Acid Cleanup	3665A	Oil & Grease	1664A
GC/Svoa/Metals TCLP Prep	1311	Dissolved Oxygen	SM45000 G
GC/Svoa/Metals SPLP Prep	1312		
Pesticides/PCB	608	Nitrogen (Ammonia)	SM4500NH3 B&G
Pesticides	8081A	Total Kjeldahl Nitrogen	351.2
PCB's	8082	Nitrogen – Nitrate/Nitrite	353.2
DRO(Diesel Range Organics)	8015B	Organic Nitrogen	351.2
CTETPH	CTETPH	Paint Filter Liquids test	9095A
GC/MS Svoa	625	Phenolics	420.4
GC/MS Svoa	8270C	Phenolics	9066
Metals Prep – Water	3010A	Phopsphorus	SM4500 PB.5 & E
Metals Prep - Soil	3050B	Orthophosphate	365.3
Metals Analysis	200.7	pH	SM4500H+B
Metals Analysis	200.8	Filterable Residue (TDS)	SM2540C
Metals Analysis	6010B	Non-Filterable Res (TSS)	SM2540B
Metals Analysis	6020	Total Residue (TS)	SM2540B
Mercury - Water	245.1	Total Volatile Residue (TVS)	160.4
Mercury - Water	7470A	Setteable Matter, Residue	SM2540F
Mercury – Soils	7471A	Total Fixed & Vol solids	SM2540G
Hardness	SM2340B	Salinity	SM2520B
Alkalinity	SM2320B	Sulfide	SM4500 S2 E
Anions	300.0	Temperature	SM2550B
Anions	9056	TOC	SM5310C
BOD/CBOD	SM5210B	TOC	9060
		ТОС	Lloyd Kahn

# Appendix 4.

# Laboratory Test Methods

Analysis	Method	Analysis	Method
GC/MS Volatiles Prep	5030B	COD	410.4
GC/MS Volatiles Prep	5035H	Total Residual Chlorine	SM4500CL G
GC/MS Volatiles Prep	5035L	Hexavalent Chromium	7196A
GC/MS Volatiles Prep	3585	Trivalent Chromium	SM3500 CR D
GC/MS Volatiles-TCLP	1311ZHE	Color	SM2120B
GC/MS Volatiles	624	Specific Conductance	120.1
GC/MS Volatiles	8260B	Specific Conductance	SM2510B
GC/MS Volatiles	OLM3.2	Cyanide (Amenable)	SM4500 CN G
GC/MS Volatiles	OLM4.3	Cyanide (Total)	335.4
GC/MS Volatiles	524.2	Cyanide (Total & Amenable)	9012B
GC/Svoa Prep - Waters	3510C	Free Cyanide	D4282 02
GC/Svoa Prep - Soils	3550B	CLP Cyanide	ILM4.0
GC/Svoa Prep - Soils	3541	Cyanide Prep – oils/soils	9013
GC/Svoa Prep - Oils	3580A	Ignitability	1030
Florisil Cleanup	3620B	Flashpoint	1020A
Sulfur Cleanup	3660B	Odor	140.1
Acid Cleanup	3665A	Oil & Grease	1664A
GC/Svoa/Metals TCLP Prep	1311	Dissolved Oxygen	SM45000 G
GC/Svoa/Metals SPLP Prep	1312	Nitrogen (Ammonia)	350.1
Pesticides/PCB	608	Nitrogen (Ammonia)	SM4500NH3 B&G
Pesticides	8081A	Total Kjeldahl Nitrogen	351.2
PCB's	8082	Nitrogen – Nitrate/Nitrite	353.2
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CTETPH	CTETPH	Paint Filter Liquids test	9095A
GC/MS Svoa	625	Phenolics	420.4
GC/MS Svoa	8270C	Phenolics	9066
Metals Prep – Water	3010A	Phopsphorus	SM4500 PB.5 & E
Metals Prep - Soil	3050B	Orthophosphate	365.3
Metals Analysis	200.7	pH	SM4500H+B
Metals Analysis	200.8	Filterable Residue (TDS)	SM2540C
Metals Analysis	6010B	Non-Filterable Res (TSS)	SM2540B
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Mercury - Water	7470A	Setteable Matter, Residue	SM2540F
Mercury – Soils	7471A	Total Fixed & Vol solids	SM2540G
Hardness	SM2340B	Salinity	SM2520B
Alkalinity	SM2320B	Sulfide	SM4500 S2 E
Anions	300.0	Temperature	SM2550B
Anions	9056	TOC	SM5310C
BOD/CBOD	SM5210B	ТОС	9060
		ТОС	Lloyd Kahn



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# Title: Mercury Analysis for Water and Wastewater using EPA 245.1 and SW846 7470A; Mercury in Drinking Water using EPA245.1; Leeman Mercury Analyzer (Cold Vapor Technique)

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## 1.0 Scope and Application

# 1.1. <u>Analytes, Matrix(s), and Reporting Limits</u>

EPA Method 245.1 and SW846 Method 7470A are applicable to the determination of mercury in water matrices. Mercury may be found in water in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique.

The typical detection limit using a 30 ml sample size is 0.1 ug/L Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

## 2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

## 3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

### 4.0 Interferences

- **4.1** The addition of potassium persulfate during the digestion step can eliminate the possible interference from sulfide in the sample without affecting the recovery of inorganic mercury.
- **4.2** Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.
- **4.3** Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 7.5 ml hydroxylamine sulfate.

### 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may

involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum. All questions pertaining to any safety procedure should be brought to the department manager or TA Edison Safety Officer.

## 5.1 Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

## 5.2 **Primary Materials Used**

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
a)Mercury b)(1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M <sup>3</sup> Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M <sup>3</sup> -TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

Material (1)		Exposure			
	Hazards	Limit (2)	Signs and symptoms of exposure		
Potassium Permanganate	Oxidizer	5 Mg/M³ for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.		
1 – Always add acid	1 – Always add acid to water to prevent violent reactions.				
2 – Exposure limit re	2 – Exposure limit refers to the OSHA regulatory exposure limit.				

# 6.0 Equipment and Supplies

# 6.1 Instrumentation

- 6.1.1. Leeman Laboratories Inc. Hydra AA Automated Hg Analyzer
- 6.1.2. Computer and Printer with Leeman WinHg software
- **6.1.3.** Block digestor (Environmental Express or SCP Science): Adjustable and capable of maintaining a temperature of 90 -95°C.

# 6.2 Supplies

- 6.2.1. 50 ml Hot Block Digestion Cups
- 6.2.2. 100 ml graduated cylinder
- 6.2.3. Eppendorf Pipettes and tips in various sizes
- 6.2.4. 100 ml volumetric flasks
- **6.2.5.** 15 ml sample cups
- 6.2.6. 8 liter carboy container
- 6.2.7. Pump tubing:
  - Sample, viton, blue tab
  - Reductant, red tab
  - Drain, blue tab
  - Rinse, black tab

- **6.2.8.** Drying Tube Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.
- 6.2.9. Nitrogen or Argon supply capable of producing 80 PSI.

# 7. <u>Reagents and Standards</u>

# 7.1. <u>Reagents</u>

Storage requirements: store at room temperature

<u>Life of Reagent:</u> - Concentrated acids: refer to manufacturer's instructions - Laboratory prepared reagents and diluted acids: one year

- **7.1.1** Sulfuric acid Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.
- 7.1.2 Nitric acid Concentrated (Trace Grade or Equivalent)
- 7.1.3 Hydrochloric acid-Concentrated (Trace Grade or Equivalent)
- **7.1.4** Potassium Permanganate (ACS Grade); for stability information, refer to manufacturer's instructions.
- **7.1.5** Sodium Chloride (analytical reagent grade); for stability information, refer to manufacturer's instructions.
- **7.1.6** Hydroxylamine Hydrochloride (ACS Grade); for stability information, refer to manufacturer's instructions.
- **7.1.7** Stannous Chloride (ACS Grade); for stability information, refer to manufacturer's instructions.
- 7.1.8 Deionized water 18 megohm minimum
- **7.1.9** 10% Hydrochloric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 800 ml of concentrated HCl and bring the final volume up to 8 liters with deionized water.
- **7.1.10** 10% Stannous chloride solution Add 50 g of SnCl₂ to 500 ml 10% HCl solution.
- **7.1.11** Sodium chloride/Hydroxylamine Hydrochloride solution Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water.
- **7.1.12** Potassium Permanganate (KMnO<sub>4</sub>) 5% solution w/v Dissolve 100 g of KMnO<sub>4</sub> in deionized water and dilute to 2 liters using deionized water.

**7.1.13** 0.15% Nitric Acid- Add approximately 5 liters of deionized water into an 8 liter carboy container. Cautiously add 12mL of concentrated HNO<sub>3</sub> and bring the final volume up to 8 liters with deionized water.

# 7.2 Standards

Storage requirements Shelf-life:	all standards are stored at room temperature Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily
	Working standards – made fresh daily
	(Note: expiration date must not go beyond the expiration
	date of the source stock).
Concentration:	see Attachment 1 for example certificates of analysis (COA) for all of the standards mixes listed below. The COA lists the manufacturer's part number, certified concentration and shelf life.

Document standard preparation in the TestAmerica Edison Mercury Standard Preparation Logbook

- **7.2.1** Stock Mercury Calibration (10 ppm Hg) Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.2.2.** Stock Mercury Quality Control Standard (10 ppm Hg) Purchase from Inorganic Ventures; store at room temperature; for stability information, refer to manufacturer's instructions. This stock standard must be purchased from a second source vendor.
- **7.2.3.** Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Stock Mercury Calibration (Sec 7.2.1) to 100 ml with 0.15% HNO<sub>3.</sub>The resulting solution will contain 100ppb Hg.
- **7.2.4.** Intermediate Quality Control Standard (DQCS-Int): Dilute 1 ml of Stock Mercury Calibration Verification Standard (Sec 7.2.2) solution to 100 ml with 0.15% HNO<sub>3</sub>. The resulting solution will contain 100ppb Hg.
- 7.2.5. Calibration Standard Preparation: Use six 100 ml volumetric flasks to prepare the standards. Add small portion of 0.15% HNO<sub>3</sub> to each flask. Working in increasing order, spike the appropriate flasks with 0.0, 0.1, 1.0, 2.0, 5.0, and 10.0 ml of working solution DCAL-Int (Sec 7.2.3). Bring to final volume of 100 ml and mix thoroughly. The corresponding concentrations are 0.0ppb, 0.1ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury respectively. For drinking water analysis, the 2.0ppb standard is also analyzed as the Maximum Contaminant Level (MCL) standard.
- **7.2.6.** Quality Control Standard (QCS) Preparation: Add a small portion of 0.15% HNO<sub>3</sub> to a 100ml volumetric flask and spike 5 ml of DQCS-Int (Sec 7.2.4).

Bring up to final volume and mix thoroughly. The resulting solution will contain 5.0 ppb of Hg.

- **7.2.7.** Initial and Continuing Calibration Verification (ICV and CCV) standards 5.0ppb: The 5.0 ppb prepared from the calibration standards (Sec 7.2.5) is used as the Initial and Continuing Calibration Verification standard (CCV).
- **7.2.8.** Maximum Contaminant Level (MCL) standard 2.0 ppb for drinking water analysis: The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard.

## 8.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>2</sup>	Reference
Waters	P. FP, G <sup>1</sup>	500 ml	HNO <sub>3</sub> , pH < 2; Cool 4±2°C	28 Days	40 CFR Part 136.3

<sup>1</sup> Polyethylene, fluoropolymer, glass

<sup>2</sup> Inclusive of digestion and analysis

# 9.0 Quality Control

**9.1.** <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Preparation Blank (PB)	1 in 20 or fewer samples	<b>245.1</b> : < MDL
		<b>7470A</b> : < MDL; <b>or;</b> < 5% of the reg limit; <b>or,</b> < 5% of the measured sample concentration (whichever is greater)
Laboratory Control Sample (LCS)	1 in 20 or fewer samples	<b>245.1</b> : 85-115% <b>7470A</b> : 80-120%
Matrix Duplicate (DUP) <sup>1</sup>	1 in 20 or fewer samples	20% RPD
Matrix Spike (MS) <sup>1</sup>	1 in 20 or fewer samples	<b>245.1</b> : 70-130% <b>7470A</b> : 75-125%
Serial Dilution (L) for 7470A	1 in 20 or fewer samples	±10%

<sup>1</sup> The sample for DUP and MS are randomly selected, unless specifically requested by a client. Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

- **9.1.1 Prepar ation Blank/Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. For 7470A, results must be less than: MDL, 5% of the regulatory limit for that analyte, or 5% of the measured concentration in the sample, whichever is greater. For 245.1 results must be less than the reporting limit. If the analyte concentration in the blank is above this control limit, the batch must be prepared again and the samples reanalyzed.
- 9.1.2 <u>Laboratory Control Sample (LCS)</u>: A laboratory control sample must be analyzed with each batch of samples digested. A blank is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. Results of the aqueous LCS must fall within ±15% of the true value for Method 245.1 and ±20% for Method 7470A.
- **9.1.3** <u>Matrix Duplicate (DUP):</u> A duplicate is analyzed for each batch of samples digested. The relative percent difference between the results of the sample and the duplicate must fall within 20 % Relative Percent Difference (RPD) for samples greater than five times the reporting limit.
- **9.1.4** <u>Matrix Spike (MS):</u> A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (0.30 ml of standard DCAL-Int). This is equivalent to 1.0 ppb Hg if a 30 ml portion of sample is digested. A recovery of 70-130% for Method 245.1 and 75-125% for Method 7470A is required.
- **9.1.5** Serial Dilution (L): For method 7470A, a five fold serial dilution must be performed on one sample per batch. Identify the sample with suffix 'L' in the run log. The sample should contain a sufficiently high concentration; minimally a factor of 25 times above the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. Results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

# 9.2 Instrument QC

**9.2.1.** <u>Initial Calibration Verification (ICV):</u> Initial calibration is verified after calibration. The ICV solution should be prepared from the same CAL standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the ICV; see Sec 7.2.7 for preparation instructions. For 245.1, the results must not differ from the true value by more than 5%. For 7470A, the results must be within 10% of the

true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

**9.2.2.** Continuing Calibration Verification (CCV): Calibration verification is performed after the calibration, after every 10 samples, and at the end of the run. The CCV solution should be prepared from the same CAL standard as used to prepare the calibration solutions. Use a concentration of mercury at the midpoint of the calibration range (5.0ppb). The Cal standard containing 5ppb is analyzed as the CCV.

For 245.1, CCV must not differ from the true value by more than 10%. For 7470A, CCV values must be within 20%. If not, stop the analysis and recalibrate. Re-analyze the previous ten samples following the last good calibration verification.

- **9.2.3.** <u>Initial and Continuing Calibration Blank (ICB/CCB):</u> ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. For methods 245.1 and 7470A, the absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.
- **9.2.4.** <u>Maximum Contaminant Level (MCL):</u> For drinking water analysis, one MCL standard shall be analyzed per calibration. The 2.0ppb standard prepared from the calibration standards (Sec 7.2.5) is analyzed as the Maximum Contaminant Level (MCL) standard. The result must be within 50% of the true value. If it's outside of the acceptable limit, terminate the analysis, correct the problem, and recalibrate the instrument.
- **9.2.5.** Quality Control Standard (QCS): The calibration is verified after calibration using a second source vendor; see Sec 7.2.6 for preparation instructions. For 245.1 and 7470A, the results must not differ from the true value by more than 10%. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.

# 10.0 Procedure

### 10.1 <u>Sample Preparation</u>

- **10.1.1** Transfer 30 ml sample (DI water for PBW and LCSW) or standard, or an aliquot diluted to 30 ml, to an appropriately identified 50 ml hot block digestion cup. For QA samples, transfer 3 aliquots of 30 ml sample to three digestion cups labeled as SAMPLE, DUP and MS. Spike LCSW and MS with 0.1 μg of mercury (0.3 ml of DCAL-INT standard).
- **10.1.2** Add 1.5 ml concentrated  $H_2SO_4$  and 0.75 ml concentrated HNO<sub>3</sub> mixing well after each addition.

- **10.1.3** Add 4.5 ml of potassium permanganate solution to each bottle. Mix well and let stand for 15 minutes (minimum); if the color has disappeared, add additional KMnO<sub>4</sub> until the purple color persists for at least 15 minutes (document in the Sample preparation log any additional amount of KMnO<sub>4</sub> added). The same amount of KMnO<sub>4</sub> must be added to the standards and samples.
- **10.1.4** Add 2.4 ml potassium persulfate solution to each bottle.
- **10.1.5** Heat for 2 hours in a 95<sup>o</sup> C hot block digestor. Remove from block digestor and cool.
- **10.1.6** Add 1.8 ml Sodium chloride Hydroxylamine hydrochloride solution to reduce the excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride Hydroxylamine HCI solution may be added. Wait at least 30 seconds after decolorization before analyzing.

## 10.2 <u>Calibration</u>

- 10.2.1. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the Standard Preparation Logbook located in the Mercury analysis room.
- **10.2.2.** Prepare five calibration standards, blank and calibration verification standard as detailed in sections 7.2.5 and 7.2.6.

### 10.3 <u>Sample Analysis</u>

- **10.3.1** Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.
- **10.3.2** The following analytical run sequence is currently used:

Instrument Calibration (Blank and five standards)

ICV ICB QCS MCL (for drinking water analysis) 9 Samples CCV CCB 10 Samples

CCV CCB Repeat until run is completed CCV CCB

- **10.3.3** Instrument Operation:
  - **10.3.3.1.** Turn on Computer, Printer and Monitor.
  - **10.3.3.2.** Plumbing the Reagent Lines:
    - **10.3.3.2.1.** One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.
    - **10.3.3.2.2.** Reductant (Red): Connect tab end of tube to the reductant tubing that is connected to the reductant bottle and the other end to the mixing tee.
    - **10.3.3.2.3.** Sample (Blue): Connect tab end of tube to the autosampler probe and the other end to the mixing tee.
    - **10.3.3.2.4.** Drain (Blue): Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.
    - **10.3.3.2.5.** Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.
  - **10.3.3.3.** Preparation of Reagents:
    - **10.3.3.3.1.** Pour the 10% SnCl<sub>2</sub> solution into the reductant bottle.
    - **10.3.3.2.** Pour the 10% HCl solution into the rinse reservoir bottle.
  - **10.3.3.4.** Start the Program:
    - **10.3.3.4.1.** Double click WinHg icon on the desktop. This will open the WinHg Runner 1.5 window.
  - **10.3.3.5.** Select the Protocol/Method:
    - **10.3.3.5.1.** Go to the Protocol box in the upper left hand corner

and click on the "▼" button.

- **10.3.3.5.2.** Scroll down to the desired protocol and click on it, (i.e. 245\_7470).
- **10.3.3.6.** Create a New Data Set/File Name:
  - **10.3.3.6.1.** From the drop down file menu, click on 'New Data Set'. Type in name of Data Set (i.e. 15990HG1), and click Ok, this opens new batch name box. Type in Batch name (i.e. 15990HG1), and click OK. The new Data Set and batch name should now appear in the respective locations.
- **10.3.3.7.** Open the Data Base Window:
  - 10.3.3.7.1. From the tool bar click on the "DB↓" button. Check to ensure that the correct protocol is open in the Protocol Box (in upper left corner) of the Data Base window. The Dataset/Protocol in the upper right hand corner, should match that in the Runner Window, (i.e. 5990HG1/DW200). Report specs should also have the same protocol open.
  - **10.3.3.7.2.** Left click the Apply button. Left click the Cal Curve tab to view calibration values.
- **10.3.3.8.** Turn on Instrument (Lamp/Gas/Pump):
  - **10.3.3.8.1.** Return to the Win Hg Runner window by clicking anywhere inside of runner window.
  - **10.3.3.8.2.** In main 'WinHg Runner' window, click on Control tab. Under Lamp click "ON" to turn lamp on. Lamp should warm up for approximately 5 minutes before analyzing any samples or standards.
  - **10.3.3.8.3.** Under Pump click "ON" to turn pump on. Click on "Standby" to keep lamp on while instrument is not running, otherwise lamp will automatically shut off after 15-20 minutes. Before analysis, click the "On" button for the pump and gas.
- 10.3.3.9. Setting up rack:
  - **10.3.3.9.1.** In 'WinHg Runner' window, click on icon that looks like rack. This will open up the rack editor.
  - **10.3.3.9.2.** Type in the Sample IDs under the "Extended ID" column, starting with sequence 1 and follow down until sequence 44. The dilution factor for the sample

is typed in under the "Extended ID" column. "Weight" and "Volume" column should always have a value of 1.00 for all samples; the actual weight and volume will be included later in the calculation and data processing. The last column is the macro column. This is where the analyst tells the instrument to automatically go to the check standard cups (i.e. C3 C1 C2 CP). Click on "File", "Save As;" use the prep batch number as the save file name.

Cup	SampleID	ExtendedID	Weight	Volume	Macros
1	PBW011505		1.00	1.00	C3 C1 C4 CP
2	LCSW		1.00	1.00	
3	610544		1.00	1.00	
4	610544D		1.00	1.00	
5	610544MS		1.00	1.00	
10	610545		1.00	1.00	C2 C1

- The Macro command C3 C1 C4 CP tells the instrument to run the check standards before running the sample cup.
- The Macro command C2 C1 tells the instrument to run the sample cup first then the check standards. Make sure the CCV/CCB is run so no more than 10 samples run between check standards.
- **10.3.3.10.** Activating and Running Standards:
  - **10.3.3.10.1.** Click on the Standards tab in the Runner Window.
  - **10.3.3.10.2.** Click on button to activate standard cup S1(0.0ppb), S2(0.1 ppb), S3(1.0ppb), S4(2.0ppb), S5(5.0ppb), S6(10.0ppb) and Rep1. Fill cups appropriately.
  - **10.3.3.10.3.** Click on C1<CCB>, C2<AICV>, C3<ACCV>, C4<ACCV>.
  - 10.3.3.10.4. Click on New Cal Reset.
  - **10.3.3.10.5.** Click on Standard Auto to run standards.
  - **10.3.3.10.6.** Check the calibration curve in the 'Database' window. If the correlation coefficient (Rho) of the calibration is ≥0.995, it is acceptable; Click Accept. Recalibrate if Rho is <0.995.
  - **10.3.3.10.7.** Print the calibration curve by hitting the printer icon.
  - **10.3.3.10.8.** In the 'WinHg Runner 1.5' window, open File from the drop down menu, choose Page eject, to print the standards on their own separate page.

- 10.3.3.11. Analyzing Samples:
  - **10.3.3.11.1.** Click on the Sample tab in the 'WinHg Runner 1.5' window.
  - **10.3.3.11.2.** Select the rack you want to run by clicking the "▼" button for Rack Name and highlighting the desired rack, for station 1 and station 2. If you're only running one rack, make sure you highlight the blank space at the top of the rack list for the unused station.
  - **10.3.3.11.3.** Enter the start cup and the end cup values. If you're only running one rack, make sure the start and end cup are blank for the empty station.
  - **10.3.3.11.4.** Real time print should be checked.
  - 10.3.3.11.5. Pour samples into polypropylene test tubes and place them in the appropriate sequence noted on rack. Rack sequence is from right to left i.e. (.... 3, 2, 1).
  - **10.3.3.11.6.** Click Run Auto to begin analyzing samples.
- 10.3.3.12. Printing Report and Post-Run Report:
  - **10.3.3.12.1.** In the 'WinHg Runner 1.5' window, click File, Page eject to print out last page of data.
  - **10.3.3.12.2.** In the 'WinHg Data Base' window, click on the "Report tab."
  - **10.3.3.12.3.** Mark the box next to the Batch ID with a check (✓) by clicking on the box. The sample IDs should now show up under the Record ID column.
  - 10.3.3.12.4. Select the IDs you wish to print by checking on the box in the far left column of the desired sample, a check mark (✓) on the box will include data in report, a box without a checkmark will exclude the sample data from the report. You can select all of them by checking the ALL button, or only a few by checking the NONE button and then clicking the box next to the desired sample.
  - **10.3.3.12.5.** Under the Report Specs, click the "▼" button to the right of the box and select the report format you wish to use.

e.g. 245\_7470 = results in concentration, and 245\_INT = results in intensities

- **10.3.3.12.6.** Click the include button under the Select Records heading.
- **10.3.3.12.7.** Click the Generate Report button at the lower right hand corner. This will activate the 'Generate Report' window.
- **10.3.3.12.8.** To send report to the printer, choose the 'report option', under the format heading along with the printer option under 'destination.' Click Generate Tab.
- 10.3.3.13. Creating a PRN File:
  - **10.3.3.13.1.** Choose the PRN option under format.
  - **10.3.3.13.2.** Choose the Disk File option under destination (should default to this option).
  - **10.3.3.13.3.** The output file path and name is: F:\INORG\LEEMAN#\DATASET NAME. You must delete C:\ before typing in path. Click Generate Tab.
- **10.3.3.14.** Shutting Down the Instrument:
  - **10.3.3.14.1.** Close the 'WinHg Database' window.
  - **10.3.3.14.2.** Open the control tab in the 'WinHg Runner 1.5' window.
  - **10.3.3.14.3.** Click the "Off" button for the lamp, pump, and gas.
  - **10.3.3.14.4.** Close the WinHg Runner 1.5 window.

### 11.0 <u>Calculations / Data Reduction</u>

**11.1** Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100 spiked concentration

**11.2** Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

**11.3.** Final results calculation in aqueous samples : Concentration = mg/ L =  $C \times V1 \times D$ 

Where:

- C= Element concentration from
  - instrument (ppb)
- V1= Final volume of sample digested (in liters)
- D= Dilution performed on sample
- V2= Initial volume of sample digested (in liters).
- **11.4.** Data Processing:
  - **11.4.1.** All data is recorded directly in TALS' Analyst Desktop II program.
  - **11.4.2.** Record standard preparations in TALS Reagent module.
  - 11.4.3. Import Data from WinHG Leeman to TALS
    - 11.4.3.1. Start WinHg and Select 'DataBase'
    - **11.4.3.2.** Select Report Tab and Batch ID
    - **11.4.3.3.** Select 'Generate Report,' and PRN File format
    - **11.4.3.4.** Select Output File Path under c:\Hg\RawData\TodaysDate.PRN
    - 11.4.3.5. Select 'Generate'
  - **11.4.4.** Metals Data Review checklist (EDS-WI-007) must be filled out prior to data submission.

# 12. Method Performance

# 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

# 12.2 Instrument Detection Limit:

The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. For 245.1, the IDL is determined by multiplying the average of the standard deviations obtained from the analysis of 10 reagent blanks by 3. For 7470A, the IDL is determined by multiplying the average of the standard deviations obtained for multiplying the average of the standard deviations of 7 reagent blanks by 3.14

# 12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

# 12.4 <u>Training Requirements</u>

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

# 13. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

# 14. Waste Management

- **14.1.** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).
- **14.2.** The following waste streams are produced when this method is carried out.
  - Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.
  - Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor. Onyx Profile WIP Number: 590598

Teris Profile Number 50016653

### 15. <u>References / Cross-References</u>

- **15.1.** <u>Determination of Mercury in Water by Cold Atomic Absorption Spectrometry,</u> EMSL-Cincinnati, EPA/600/R-94/11, May 1994; Method 245.1 Revision 3.0.
- **15.2.** <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- **15.3.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.4.** TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.
- **15.5.** TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision.
- **15.6.** Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.
- **15.7.** Metals Data Review Checklist Work Instruction # EDS-WI-007, most current revision.
- **15.8.** Leeman Hydra AA Operating Manual

### 16. Method Modifications:

ltem	Method No.	Modification
Sample Preparation	SW 7470A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.
Sample Preparation	SW 7470A EPA 245.1	The hotblock has replaced the hot-water bath for digestion. This modification has been made to reduce cross-contamination (the hotblock tubes are disposable).
Sample Preparation	SW 7470A EPA 245.1	The typical prep sample size is reduced to 30ml (previously 100ml). This modification was made to allow for limited available sample volumes. Reagent volumes were adjusted to maintain sample to reagent volume ratio

### 17. <u>Attachments</u>

Attachment 1: Example Certificate of analysis (10 ppm Hg)

### 18. <u>Revision History</u>

- Revision 6, dated 03 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Combined SOP ED-MT-014 and ED-MT-015 with SOP ED-MT-017; retired SOP ED-MT-014 and ED-MT-015 at the effective date of this SOP.
  - Sec 6.1.3: Replaced water bath with block digestor.
  - Sec 6.2.1: Replaced 300 ml BOD bottles with 50 ml hot block digestion cups
  - Sec 6.2.3. Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines
  - Sec 7.1: Added 0.15% HNO3 to list of reagents. Deleted Potassium Persulfate, Magnesium Perchlorate, 0.5N H2SO4 & 5% Potassium Persulfate solution to list of reagents; Reagents deleted are not applicable to this method.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
  - Sec 7.1.9 & 7.1.13: Revised preparation procedure to reflect actual lab practices.
  - Sec 7.2.6: Renamed the second source standard 'Initial Calibration Verification standard' to 'Quality control standard.' QCS is added in the Instrument QC and analytical run sequence.
  - Section 8: Updated the section into Table format and have included the sample container, sample size requirements and method reference.
  - Changed the verification standard concentration of ICV (3ppb) to 5ppb; CCV concentration remains 5ppb.
  - Revised control limits to comply with Method 245.1
  - Sec 9.1.1: Clarified QC limits for the Preparation Blank
  - Sec 9.1.2: Revised the LCS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±15%.
  - Sec 9.1.4: Revised the MS limits for wastewater samples analyzed via Method 245.1 from ±20% to ±30%.
  - Sec 9.1.5: Added Serial Dilution (L) in Sample QC
  - Sec 9.2.1: Clarified the recovery limits of ICV for method 245.1; % Rec limits were revised from 10% to 5% to reflect actual laboratory practices.
  - Sec 10.3.2: Added MCL in the analytical run sequence.
  - Sec 10.3.3.2: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
  - Sample size reduced from 100 ml to 30ml; preparation of the LCS (Sec 9.1.2) and MS (Sec 9.1.4) were revised to reflect this change in sample volume.
  - Sec 10.1: Adjusted reagent volume to maintain sample to reagent ratios.
  - Sec 11: Updated data processing in accordance with the new TALS.
  - Sec 15: Added applicable references.
  - Sec 16: Described the elimination of stannous chloride in the sample preparation and replacement of hot water bath as a method modification.

## Attachment 1

P	INORGANIC	S	
)	Lakewood, New Jersey 08701 inorganicventures.com	usa fax: 732,901.1903 Rec (d. 1-12-09 info@inorganicventures.com	
	k .		
1.0	INORGANIC VENTURES is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."		
2.0	DESCRIPTION OF CRM	10 µg/mL Mercury in 10% (v/v) HCL	
	Catalog Number: Lot Number: Starting Material: Starting Material Purity (%): Starting Material Lot No: Matrix:	MSHG-10PPM <b>B2-HG02061</b> Hg metal 99.999549 05214TX 10% (v/v) HCL	
3.0	CERTIFIED VALUES AND UNCERTAINTIES		
	Certified Concentration:	10.027 ± 0.020 µg/mL	
·	Certified Density:	1.019 g/mL (measured at 22° C)	
. 1			
	Uncertainty $(\pm) = \frac{2[(\Sigma s)]}{(n)}$	6.45	
4.0	TRACEABILITY TO NIST	AND VALUES OBTAINED BY INDEPENDENT METHODS	

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually
national or International standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd
ed., 1993, definition 6.10)

. This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:

# Attachment 1 (continued)

Certificate of Analysis			
Catalogue Number : Description :	141-110-111/141-110-112/141-110-115 PlasmaCAL - ICP-MS Verification Standard 1 Solution B		
Lot Number : Expiration Date :	SC8298812 January 2010		
Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133			
Actual Concentrations			
н	ig : 9.98 μg/ml		
Matri Dens			
MS 0882			
MS 0882 Rec d 1/14/09			
Certified by :	Certification Date : October 28, 2008		
This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid- cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)			
21800 Clark Grah Phone : (51	D 9001:2000 Quality System and ISO 17025 (in-process) SCP SCIENCE nam, Baie D'Urfé, QC, Canada H9X 486 (4) 457-0701 Fax: (514) 457-4499 Site: www.scbscience.com		



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# Title:Trace Metals Analysis by Inductively Coupled PlasmaEmission Spectroscopy by SW846 Method 6010B

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	Approvals (	Signature/Date):	
Donald Evans Donald Evans Department Manager	<u>04/25/11</u> Date	Kene' Kasperek Health & Safety Manage	04/25/11 Date er / Coordinator
Candon Carl Armbruster Quality Assurance Manager	04/22/11 Date	Ann Gladwell Laboratory Director	<u>04/22/11</u> Date

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#### 1.0 Scope and Application

#### 1.1. <u>Analytes, Matrix(s), and Reporting Limits</u>

Method 6010B determines trace elements in solution using inductively coupled plasma-atomic emission spectrometry (ICP-AES). The method is applicable to all of the elements listed in Table 1. All matrices excluding filtered groundwater samples but including aqueous samples, TCLP/SPLP extracts, soils, sludges, and sediments, require some type of digestion step prior to analysis (i.e. 3050B, 3010A & 3005A).

Detection limits, sensitivity, and the optimum and linear concentration ranges of the elements can vary with the wavelength, spectrometer, matrix and operating conditions. Elements listed in Table 1 may be analyzed by this method if performance at the concentration levels of interest is demonstrated. The laboratory's reporting limits (RL) are listed in Table 1.

Element	Aqueous Reporting Limit (Non-TCLP) (ug/L)	Aqueous Reporting Limit (TCLP) adjusted for typical 5X-dilution (ug/L)	Soil Reporting Limit, adjusted for typical 4X-dilution (mg/Kg)
AI	200	1000	40
Sb	10	50	2
As	5	25	1
Ва	200	1000	40
Be	2	10	0.4
Во	50	250	10
Cd	5	25	1
Са	5000	25000	1000
Cr	10	50	2
Со	50	250	10
Cu	25	125	5
Fe	150	750	30
Pb	5	25	1
Mn	15	75	3
Mg	5000	25000	1000
Мо	20	100	4
Ni	40	200	8
K	5000	25000	1000
Se	10	50	2
Ag	10	50	2
Na	5000	25000	1000
Sr	20	100	4
TI	10	50	2
Sn	50	250	10
Ti	20	100	4
V	50	250	10
Zn	30	150	6

#### Table 1: Reporting Limit for Soil and Water

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 2.0 <u>Summary of Method</u>

Samples are digested with the appropriate digestion technique prior to sample analysis. SW846 Method 3050B (TestAmerica Edison SOP No. ED-MTP-005) is the procedure utilized for digesting metals in soil samples; SW846 Method 3010A (TestAmerica Edison SOP No. ED-MTP-003) and SW846 Method 3005A (TestAmerica Edison SOP No. ED-MTP-002) are the procedures utilized for digesting metals in water samples Following the sample preparation/digestion, a multi-elemental analysis of elements in solution is performed via Inductively Coupled Plasma- Atomic Emission Spectrometry (ICP-AES). Elemental constituents are determined simultaneously. Samples are nebulized and the aerosol is transported to the plasma torch where excitation occurs. Element specific spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed by a grating and the intensities are measured. Background correction points and/or inter-element correction factors must be determined at instrument and method set-up. This laboratory semi-annually investigates the need for such factors and copies of the data are available. The points and correction factors determined are used throughout the year and verified on a daily basis. If a discrepancy exists, the situation is remedied.

#### 3.0 <u>Definitions</u>

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 4.0 Interferences

- **4.1.** Most interference are eliminated or greatly reduced during the acid digestion of the sample matrix. High salt concentrations can cause analyte signal suppressions and confuse interference tests. Dilute the sample if necessary.
- **4.2.** Spectral interferences encountered on the instrument are corrected for by using baseline correction points and/or by applying background correction factors. Background correction points are determined by scanning the area on either side of the wavelength and recording the apparent intensity from all other method analytes. Use single element solutions at or near the upper linear range of each element. Interelement Correction Factors are determined by analyzing single element solutions and recording the apparent analyte concentration. All interfering elements must be analyzed at the same time as the elements of interest.
- **4.3.** Physical interferences are effects associated with sample nebulization and transport. They are reduced or eliminated with use of a peristaltic pump and internal standards. If these methods are insufficient to reduce the interference, they must be reduced by diluting the sample.
- **4.4.** Memory interferences result when analytes in a previous sample contribute to the signals measured in a new sample. The necessary rinse times must be estimated prior to sample analysis. Until required rinse times are determined a suggested

rinse time of 60 seconds shall be used. If a memory interference is suspected the sample must be reanalyzed.

**4.5.** Chemical interferences include molecular compound formation, ionization effects, and solute vaporization effects. Normally, these effects are not significant with the ICP technique. If observed, changing the operation conditions (e.g., forward power) and/or buffering the sample (adding 2% Lithium to the internal standard solution) should reduce or eliminate the interference.

#### 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

#### 5.1. Specific Safety Concerns or Requirements

The ICP plasma emits strong UV light and is harmful to vision. All analysts must avoid looking directly at the plasma.

#### 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Hydrochloric Acid	Corrosive Poison	5 ppm- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Nitric Acid	Corrosive Oxidizer Poison	2 ppm- TWA 4 ppm- STEL	Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
1 – Always add a 2 - Exposure limi			nt reactions. ory exposure limit.

## 6.0 Equipment and Supplies

#### 6.1. Instrumentation

- iCAP 6500 Duo View ICP-OES Spectrometer with a solid state 27.12MHz RF generator. The nebulizer, plasma, and auxiliary gas to the sample introduction system are supplied and controlled via 3 independent mass flow controllers. Vacuum purged spectrophotometer with an axial and radial plasma torch. Purchased from ThermoFisher Scientific.
- Thermo Jarrel Ash Model 61E Trace ICP with 486 microprocessor, monitor, printer and autosampler. Resolution 0.011 nm on a holographically grooved grating (2400 grooves/mm). Vacuum purged spectrophotometer with an axial plasma torch.

Note: Operating conditions must be established by the analyst according to the instrument manufacturer's specification and must meet conditions satisfying the analytical and quality assurance requirements.

#### 6.2. <u>Supplies</u>

- Volumetric Flasks (Class A): 50 mLs, 100 mLs, 500mls & 1000mls
- Eppendorf & Fisher Pipettes, varying volumes
- Polypropylene tubes
- Argon supply 99.5%

#### 7. <u>Reagents and Standards</u>

#### 7.1. <u>Reagents</u>

- **7.1.1.** 18 megohm Reagent grade Type II water
- **7.1.2.** Concentrated distilled nitric acid--Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.1.3.** Concentrated distilled hydrochloric acid--Trace Grade or Equivalent; store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.1.4.** Reagent water, 5% HNO3 + 5% HCI: Add 1 liter of concentrated HNO3 and 1 liter of concentrated HCI to deionized water and bring to 20 liter volume with deionized water. Note: Always add acid to water. Record preparation in the ICP Reagent Dilution logbook. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature. *Note*: This reagent water is required for the analysis of digested samples.
- **7.1.5.** Reagent Water, 5% HNO3: Add 1 liter of concentrated HNO3 to deionized water and bring to 20 liter volume with deionized water. Note: Always add acid to water. Record preparation in the ICP Reagent Dilution logbook. Prepare every 12 months or refer to manufacturer's expiration date; store at room temperature. *Note*: This reagent water is required for the analysis of undigested samples.

#### 7.2. <u>Standards</u>

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions
	Intermediate standards – 12 months
	Working standards – 12 month
	(Note: expiration date must not go beyond the expiration
	date of the source stock).
Concentration:	see Attachment 3 for example certificates of analysis (COA)
	for all of the standards mixes listed below. The COA lists
	the manufacturer's part number, certified concentration and
	shelf life.

Standards must be prepared every 12 months or sooner if needed or required. "If needed" means the standard has been exhausted; "if required" means that the standard does not meet the QC criteria. All standards must be prepared in reagent water (see Sec. 7.1.4 and 7.1.5).

**7.2.1.** <u>Calibration stock standards</u> - CLPP-CAL-1, CLPP-CAL-3, STLNJ-CAL-1A, STLNJ-CAL-1B, STLNJ-CAL-2, 10000ppm Aluminum, TANJ-STD-3, and

TANJ-STD-4; purchased from Inorganic Ventures, Christiansburg, VA. See Attachment 3 for details.

- **7.2.2.** <u>Working Calibration Standards:</u> see Attachment 1 for final elemental concentration.
  - **7.2.2.1.** <u>CAL1:</u> add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of TANJ-STD-3 and TANJ-STD-4. Bring to volume with reagent water.
  - **7.2.2.2.** <u>CAL2:</u> add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 1 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 2 ml of STLNJ-CAL-2, 2.3 ml of 10000 ppm Aluminum, and 0.5 ml of CLPP-CAL-3. Bring to volume with reagent water.
  - **7.2.2.3.** <u>CAL3:</u> add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 5 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 10 ml of STLNJ-CAL-2, 11.5 ml of 10000 ppm Aluminum, and 2.5 ml of CLPP-CAL-3. Bring to volume with reagent water.
  - **7.2.2.4.** <u>CAL4:</u> add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of CLPP-CAL-1, STLNJ-CAL-1A, and STLNJ-CAL-1B, 20 ml of STLNJ-CAL-2, 23 ml of 10000 ppm Aluminum, and 5 ml of CLPP-CAL-3. Bring to volume with reagent water.
- **7.2.3.** <u>Calibration Verification Stock Standards</u> Part No. SM-606-098 (Duo\_CCV-int) Solutions: A, B, C, and D; purchased from High-Purity Standards. See attachment 1 for final elemental concentrations.
- 7.2.4. Working Calibration Verification Standards:
  - **7.2.4.1.** <u>ICV/CCV</u>: add 100 ml of reagent water to a clean 1000 ml volumetric flask. Add 10 ml each of Part No. SM-606-098 (Duo\_CCV-int) Solutions: A, B, C, and D. Bring to volume with reagent water.
- 7.2.5. Stock Spike Standards:
  - **7.2.5.1.** Solutions A, B, C, and D (ICP-Spk), Part No. 4400-100419DD01: commercially purchased from CPI International. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 3 for the standards certified concentrations and catalog numbers.
  - **7.2.5.2.** Solutions A, B, C, and D (TCLP-Spk), Part No. SM-606-114: commercially purchased from High-Purity Standards. Store at room temperature; for stability information, refer to manufacturer's instructions. See Attachment 3for the standards certified

concentrations and catalog numbers.

- 7.2.5.3. Working Spike Standards
  - 7.2.5.3.1. ME\_LCS-int (for ICP prep) Add the following to a 1000 ml volumetric flask and bring to volume using 5% HNO<sub>3</sub>: 50 ml each of Part No. 4400-100419DD01 Solutions A, B, C, and D. Record standard preparations in TALS Reagent module. Note: Standard must not exceed the earliest expiration date of any one of its components.
  - 7.2.5.3.2. ME\_TCLPspk\_(for TCLP prep) Add the following to a 200 mL volumetric flask and bring to a volume using 5% HNO<sub>3</sub>: 40 ml each of TCLP-Spk Solutions: A, B, C, and D- Part No. SM-606-114. Record standard preparations in TALS Reagent module. Note: Standard must not exceed the earliest expiration date of any one of its components.
- **7.2.6.** <u>Stock Interference Check Standards</u> –CLPP-ICS-A, IV-7, IV-19, K 10,000 ppm, Na 10,000 ppm; purchased from Inorganic Ventures, Christiansburg, VA.
- **7.2.7.** <u>Working Interference Check Standard A (ICSA)</u>: add 500 ml of reagent water to a clean 1000 ml volumetric flask. Add 100 ml of CLPP-ICS-A stock standard solution. Bring to volume with reagent water. See Attachment 1 for final elemental concentration.
- **7.2.8.** <u>Working Interference Check Standard AB (ICSAB)</u>: add 500 ml of reagent water to a clean 1000 ml volumetric flask. Add 100 ml CLPP-ICS-A, 1 ml each of IV-7, IV-19, and LCSW-III (Sec 7.2.8.1), 0.9 ml each of 10000 ppm Sodium and 10000 ppm Potassium. Bring to volume with reagent water. See Attachment 1 for final elemental concentration.
  - **7.2.8.1.** LCSW-III: add 100 ml of 5% HNO<sub>3</sub> to a clean 200 ml volumetric flask. Add 18 ml of 10,000 ppm Sodium; add 2 ml each of 10,000 ppm Tin and 10,000 ppm Strontium. Bring the volume with 5% HNO<sub>3</sub>.
- **7.2.9.** <u>Interferent-10 (INT-10)</u>: add 500 ml of reagent water to a clean 1000 ml volumetric flask. Using 10000 ppm standards, add the following: 1 ml each of Mn, Ti, Cr, Sr, Sn, Co, and Ni; add 0.5ml each of V and Mo. Bring to volume with reagent water.

	erent-10 (INT-10) concentrations (mg/L)
ELEMENT	
Chromium	10
Cobalt	10
Nickel	10
Titanium	10
Strontium	10
Tin	10
Manganese	10
Vanadium	5
Molybdenum	5

#### 8. <u>Sample Collection, Preservation, Shipment and Storage</u>

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>1</sup>	Reference
Waters	Plastic, glass	100 mLs	HNO <sub>3</sub> , pH < 2 prior to shipment; if not, acidify upon receipt in lab <sup>2,3</sup>	180 days	SW846 Method 6010B 40 CFR Part 136.3
Soils	Plastic, Glass	3 grams	Cool 4 ± 2°C	180 days	SW846 Method 6010B

<sup>1</sup> Inclusive of digestion and analysis.

<sup>2</sup> Acid preservation may be omitted for shipping; however, acid must be added upon receipt in the lab. Following acidification, mix the sample and hold for at least 24 hours. Just prior to digestion or direct analysis, verify pH<2. If pH≥ 2, repeat steps (i.e., add acid, hold for 24hrs, verify ph<2).</p>
<sup>3</sup> Aqueous samples may be stored at room temperature.

Note: Samples for dissolved metals should be filtered in the field before acid is added to the sample. If the sample is to be filtered in the lab, no preservative is added to the sample until the sample is filtered.

#### 9. Quality Control

**9.1.** <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

Quality Control	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	<mdl; or<br="">&lt;10% of RL; or &lt;10% of Reg. Limit; or &lt;10% of the measured concentration in the sample</mdl;>
Laboratory Control Sample (LCS) for aqueous samples	1 in 20 or fewer samples	80-120%
Laboratory Control Sample (LCS) in soil	1 in 20 or fewer samples	Vendor's certified limits
Matrix Duplicate <sup>1</sup>	1 in 20 or fewer samples	If original sample and dup are both $\ge 5X$ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS) <sup>1</sup>	1 in 20 or fewer samples	75-125%
Serial Dilution (1/5 dilution) <sup>1</sup>	1 per batch of 20 or fewer samples	See sec 9.1.7.
Post Digestion Spike <sup>1</sup>	1 per batch of 20 or fewer samples	75-125%

<sup>1</sup> The sample for MS is randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

<sup>2</sup> Statistical control limits are updated annually and are updated into lab database.

- **9.1.1** Method Blank (MB): One laboratory method blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). The method blank is used to identify possible contamination during acid digestion. Results must be less than the highest of: (1) MDL, (2) 10% of the RL, (3) 10% of the regulatory limit, (4) 10% of the measured concentration of the sample. If any analyte concentration in the blank is above the control limit, the batch must be prepared again for the element in guestion and the samples reanalyzed.
- **9.1.2.** Laboratory Control Sample (LCS): A laboratory control sample must be analyzed with each group of samples digested. Results of the aqueous LCS must fall within ±20% of the true value. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the certified limits for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed. For dissolved aqueous samples not undergoing a preparation procedure, the CCV may serve as the LCS.

- 9.1.3. Matrix Duplicate (DU): A duplicate is analyzed for each batch of samples. If original sample and duplicate are both ≥ RL (reporting limit), then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.
- **9.1.4. Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples digested. A recovery of 75-125% is required. An exception to this occurs if the sample concentration exceeds the spike concentration by a factor of four or more. If the recovery is not within specified limits a post digestion spike is required to be analyzed at a concentration between 10 to 100 times the instrument detection limit. If the Post digestion spike recovery is not recovered within 75-125% a matrix effect should be suspected. See Attachment 2 for list of the elemental concentrations.
- **9.1.5. Post-Digestion Spike:** A post-digestion spike is analyzed for each batch of samples digested. Prepare the sample by mixing 0.20 ml of the working standard ME\_LCS-int (Sec. 7.2.5.3.1.) and 9.80 ml of the sample digestate. If sample requires dilution, adjust the sample as appropriate, (e.g. sample at 4X dilution, add 0.20 ml of the standard spike to 2.5 ml of sample digestate, and add 7.3 ml of the reagent water). Limits for post digestion spikes are 80-120% recovery. If both the MS and post digestion spike fail, then matrix effects are confirmed.
- **9.1.6.** Serial Dilution (SD): A 1/5 dilution is prepared and analyzed on one sample per batch to determine if matrix interferences are present.
  - **9.1.6.1.** Select a sample and dilute the digestate by a factor of 5 (DF=5).
  - **9.1.6.2.** Analyze the dilution using the same procedures used for the undiluted aliquot.
  - **9.1.6.3.** Compare the results of the diluted and un-diluted aliquots of sample digestate.
  - **9.1.6.4.** If the analyte concentration is minimally a factor of 10 above the RL (report limit), then the results of the dilution should be within  $\pm 10\%$  of the result of the undiluted sample. If not, then a chemical or physical interference effect should be suspected.

#### 9.2. Instrument QC

**9.2.1.** Initial and Continuing Calibration Verification (ICV/CCV): Instrument calibration is verified using an independent standard at the midpoint of the calibration curve. The source must be different from the calibration standards. Results must be within 10% of the true value and the replicate measurements must be less than 5% RSD. (Note: Section 8.6.1 of Method 6010B allows the use of the ICV as the CCV if the element concentrations in the solution are near the mid-point of the calibration.) If results are outside of the specified limits, terminate the analysis, correct the problem, recalibrate the instrument, and reanalyze the samples following the last good CCV. Subsequent verification is performed after every 10 samples and at the end of the analytical run.

- **9.2.2.** Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the reporting limit. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.
- **9.2.3.** Interference Check Solution A & B (ICSA/ICSAB): Verify the interelement and background corrections by analyzing the interference check solutions (ICSA & ICSAB) at the beginning of the analysis run. The ICS is analyzed in order to demonstrate that proper corrections are being utilized for known interferences. Analyst must evaluate these solutions in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the interference check solutions. Results should fall within the control limit of +/-20% of the true value or +/-2 times the RL of the true value, whichever is greater. See Attachment 1 for list of elements and the corresponding concentrations. See Sec 7.2.7 and Sec 7.2.8 for preparation instructions.
- **9.2.4.** Interferent-10 (INT-10): An INT-10 sample is analyzed for each analytical run. Results should fall within the control limit of +/-20% of the true value or +/-2 times the RL of the true value, whichever is greater; if not, problem must be identified and corrected. Analyst must evaluate this solution in order to detect trends that require corrective actions. Pay particular attention to false positives and false negatives for elements not present in the solution. Refer to Sec 7.2.9 for preparation instructions and final elemental concentrations (mg/L).

#### 10. Procedure

#### 10.1. <u>Sample Preparation</u>

Prior to sample analysis, digest samples following sample digestion procedures: aqueous samples are digested using SW846 Method 3005 or Method 3010A. Soil samples are digested using Method 3050B. Refer to TestAmerica Edison SOP Nos. ED-MTP-002, ED-MTP-003, ED-MTP-005, for the digestion procedure (see Section 15 for complete references).

- **10.2.** Set up the instrument according to the instrument's operating parameters.
  - 10.2.1. TJA 6500 Duo ICP
    - **10.2.1.1.** Set up the instrument with the operating parameters recommended by the manufacturer as specified in the iCAP 6000 Series ICP-OES Spectrometer Operator Manuals (see Sec 15.11 for reference). Allow the instrument to become

thermally stable, minimally 30min. Perform a torch alignment by aspirating a 2 ppm Zn standard.

- **10.2.1.2.** Background correction points must be determined during the initial set-up of the instrument. Refer to the iTEVA Software Manual located in the Operator Manuals for instructions.
- **10.2.1.3.** Interelement Correction factors must be verified semiannually. Refer to the iTEVA Software Manual located in the Operator Manuals for instructions. Criteria for determining IEC's is an apparent positive or negative concentration for the analyte that falls outside of one reporting limit from zero.

#### **10.2.2.** <u>TJA 61E Trace ICP</u>

**10.2.2.1.** Instrument Operating Parameters: Set up the instrument with the operating parameters recommended by the manufacturer and as specified in the instrument operation SOP, TestAmerica SOP No. ED-MT-013, The Use of Thermo Jarrell Ash Model 61E Trace Simultaneous Inductively Coupled Plasma Emission Spectrometer.

#### 10.3. Instrument Performance Criteria

Prior to the analysis of any samples the following must be performed.

- **10.3.1.** The IDL for each analyte must be determined for each wavelength used on each instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying by 3.14 the average of the standard deviations obtained from the analysis of seven replicates of a reagent blank signal.
- 10.3.2. <u>Determination of Linear Dynamic Range (LDR)</u>:

Determine the linear dynamic range of the instrument by analyzing a minimum of three high standards at the upper limit of the instrument. The analytically determined concentration of these standards must be within 10% of the true value. The linear ranges should be re-determined semiannually or if the instrument is significantly changed.

- **10.3.2.1.** Prepare the standard at concentrations that are expected to define the linear range of the instrument. The calibration standards and the linear range standards should be matrix matched; that is, they have the same percentage of hydrochloric and nitric acids.
- **10.3.2.2.** Analyze the standard(s) after the initial calibration is validated.
- **10.3.2.3.** Compare the concentration of the linear range standard with its true concentration using the following equation:

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Percent Difference = 
$$\left| \frac{Ccal - Ctrue}{Ctrue} \right| \otimes 100$$

Where:

Ccal = concentration determined from analysis Ctrue = true concentration of the standard

Note: If the percent difference is less than or equal to 10%, the linear range is confirmed at that concentration. If the percent difference is greater than 10%, repeat the analysis with a lower concentration. For elements validated in this manner you may report data up to 90% of that linear range before a dilution is required.

#### 10.4. Calibration

- **10.4.1.** Profile and calibrate the instrument according to the instrument manufacturer's instructions using a blank (reagent water) and 4 standards, see Section 7.2.2 for details on calibration standards preparation.
- 10.4.2. The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The correlation coefficient of the calibration curve must be ≥0.995. If not, the problem must be corrected, and the instrument must be recalibrated.

#### 10.5. <u>Sample Analysis</u>

- **10.5.1.** Following a sample preparation/digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any complexities, which might adversely affect the acquisition of valid data.
  - **10.5.1.1.** Soil samples and relating QC samples (except soil MB) are typically diluted 4X prior to analysis. Soil MB are diluted 2X prior to analysis.
  - **10.5.1.2.** TCLP samples and relating QC samples (except TCLP MB and LCS) are diluted 5X prior to analysis. TCLP MB and LCS are not diluted.
- **10.5.2.** Flush the instrument between standards and samples, minimally 60 seconds, using the calibration blank.
- **10.5.3.** The following analytical run sequence is currently used for samples run under the SW846 6010B protocol:

Typical Analytical Sequence
INSTRUMENT WARM-UP (approx. 30 minutes)
Align torch using 2 ppm Zn std (Duo) or profile with 5 ppm
Arsenic (Trace)
STANDARDIZATION/CALIBRATION (Blank, 4 Cal Stds)
ICV
ICB
ICSA
ICSAB
Int-10
7 Samples
CCV
ССВ
10 Samples
CCV
ССВ
10 Samples
CCV
CCB

- **10.5.4.** A minimum of two exposures for each standard, sample and blank is required. The average of the exposures is reported.
- **10.5.5.** Dilute and reanalyze all samples for which the required analytes exceed 90% of the linear range as well as samples that contain high concentrations of an interfering element.

#### 11.0. Calculations / Data Reduction

**11.1.** Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100 spiked concentration

**11.2.** Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

**11.3.** Final results calculation in soil samples: Concentration = mg/kg =  $C \times V \times D$ W

Where:

- C= Element concentration from Instrument (ppb)
- V= Final volume of sample digested (in liters)
- D= Dilution performed on sample

W= Initial dry weight of sample digested (in gram)

**NOTE:** All dry weight corrections are made in LIMS at the time the final report is prepared.

**11.4.** Final results calculation in aqueous samples : Concentration = mg/ L =  $C \times V1 \times D$ 

Where:

C= Element concentration from instrument (ppb)

- V1= Final volume of sample digested (in liters)
- D= Dilution performed on sample)
- V2= Initial volume of sample digested (in liters).
- **11.5.** Data Reduction:
  - **11.5.1.** All data is recorded directly in TALS' Analyst Desktop II program.
  - 11.5.2. Record standard preparations in TALS Reagent module.
  - **11.5.3.** Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, initial and final sample volume, spike name and amount used, all reagents and their corresponding lot numbers, creation date and expiration date.
  - **11.5.4.** Metals Data Review checklist (EDS-WI-007) must be filled out prior to data submission.

#### 12.0. <u>Method Performance</u>

#### 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

#### 12.2. Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

#### 13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

#### 14.0. Waste Management

- **14.1.** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).
- **14.2.** The following waste streams are generated as a result of this analysis:
  - Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.
  - Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor. Onyx Profile WIP Number: 590598 Teris Profile Number 50016653
  - Soil Retain Samples These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710 Onyx Profile Number: (stabilization) 402535

#### 15.0. <u>References / Cross-References</u>

- **15.1.** Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; SW-846, Method 6010B.
- **15.2.** <u>TestAmerica Edison SOP ED-MT-013</u>, The Use of the Thermo Harrell Ash Model 61E Trace Simultaneously Inductively Coupled Plasma Emission Spectrometer, most current revision.
- **15.3.** <u>TestAmerica Edison SOP ED-MTP-005</u>, *Hot Block Digestion of Sediments, Sludges and Soils, SW846 Method 3050B*, most current revision.
- **15.4.** <u>TestAmerica Edison SOP ED-MTP-003</u>, *Digestion of Water and Wastewater Samples for analysis by ICP, SW846 Method 3010A*, most current revision.
- **15.5.** <u>TestAmerica Edison SOP ED-MTP-002</u>, *Digestion of Water and Wastewater Samples for analysis by ICP, SW846 Method 3005A*, most current revision.
- **15.6.** Thermo Jarrell Ash Model 61E Trace User Manual.
- **15.7.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.8.** TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.
- **15.9.** <u>TestAmerica Corporate SOP No. CA-Q-S-005</u>, *Calibration Curves (General)*, most current revision.
- **15.10.** TestAmerica Edison Work Instruction # EDS-WI-007, Metals Data Review Checklist, most current revision.
- **15.11.** iCAP 6000 Series ICP-OES Spectrometer Operator Manuals (8499 400 90001), Thermo Electron Corporation, 2005.
- **15.12.** TestAmerica Corporate Environmental Health and Safety Manual, Document No. CW-E-M-001, most current revision.

#### 16.0. <u>Method Modifications:</u>

None

#### 17.0. Attachments

Attachment 1: Working Standard Concentration for ICP Elements in ug/L Attachment 2: Matrix spike concentration for soil and aqueous samples Attachment 3: Certificate of analysis of each multi-element stock standard

#### 18.0. <u>Revision History</u>

- Revision 7, dated 25 April 2011
  - Sec 1.1: Revised Table 1 to include the reporting limits for Aqueous TCLP/Non-TCLP and soils; included the information in Table 2 to Table 1; deleted Table 2.
     Sec 3: Updated the LQM reference for the definitions.
  - Sec 4.0: Expanded the possible interferences for this method.
  - Sec 6.1: Added iCAP 6500 Duo View ICP-OES to the list of instruments.
  - Sec 6.2: Revised to include Argon supply to the list of supplies; deleted argon supply previously listed in Sec 7.1.4. Deleted Nitrogen which is not used by this method. Subsequent sections adjusted accordingly.
  - Sec 7.1.5: Added 5% HNO3 as the reagent for the analysis of undigested samples
  - Sec 7.2.1: Added TANJ-STD-3 and TANJ-STD-4; deleted standards Zn and La
  - o Sec 7.2.2: Updated the preparation of CAL1, CAL2, CAL3, and CAL4
  - Sec 7.2.3: Added Solutions: A, B, C, and D, Part No. SM-606-098 (Duo\_CCV-int)
  - Sec 7.2.4.1: Updated the preparation of ICV/CCV
  - o Sec 7.2.5.1: Added Solutions: A, B, C, and D, Part No. SM-606-098 (Duo\_CCV-int).
  - Sec 7.2.5.2: Added Solutions A, B, C, and D, Part No. SM-606-114 (TCLP-Spk).
  - Sec 7.2.5.3.1: Updated the preparation of ME\_LCS-int.
  - Sec 7.2.5.3.2: Added the preparation of ME\_TCLPspk.
  - Deleted RepLim-INT, previously Sec 7.2.9, subsequent sections adjusted accordingly.
  - Sec 7.2.9: Replaced INT-20 with INT-10.
  - Sec 8: Added footnotes 2 & 3.
  - Sec 9: Deleted all entries of the Blank Spike (BS) and LCSD for this SOP.
  - Sec 9.1.1: Updated pass/fail criteria for the MB.
  - Deleted the following QC samples: HSA (previously sec 9.2.3), Replim and Replim2 (previously sec 9.2.6 & 9.2.7), subsequent sections adjusted accordingly.
  - Sec 9.2.4: Updated pass/fail criteria for ICSA/ICSAB
  - $_{\odot}$  Sec 10.2: Added the TJA 6500 Duo instrument set up procedure.
  - Sec 11.5: Updated Data Reduction section in accordance with TALS.
  - Sec 15.0: Added applicable references.
  - Updated Attachment 1 & 2 and removed Attachment 3 (Reporting Limt Check Standard – RepLim) and Attachment 4 (Interferent-20, Int-20). Attachment 5 (COAs) is now referred to as Attachment 3.
- Revision 1, dated June 26, 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Added Table 2, reporting limits for soil and water by method 6010B.
  - Section 2: Included applicable digestion technique for method 6010B.
  - Section 7 Reagents and Standards: Added Reagent water (5% HNO3+5% HCl) in the list of reagents; included stability and storage information of all reagents and standards.
  - Section 7.2 Standards: Added standards (ICP-PSPK, RepLim-INT & INT-20) in the list of instrument QC samples performed on this method.
  - Section 8: Updated in Table format and have included sample container, sample size requirements and method reference.
  - Section 9 Quality Control: Updated to include all Sample and Instrument QC for both matrices (soil and water).
  - Section 9.1.6, Added Post-digestion spike and defined its spiking procedure,

recovery limits and frequency.

- Section 9.2 Instrument QC: Added instrument QCs such as RepLim, RepLim-2, INT-20 and described their frequency, control limits and spiking procedure.
- Section 10 Procedure: Clarified steps performed during instrument operation including referencing TestAmerica SOP No. ED-MT-013.
- Section 15 References: Added references applicable to the SOP.
- Section 17 Attachment: Added attachments 2, 3 & 4 to list the elemental concentrations of BS, MS (for water and soil), RepLim, RepLim-2 and INT-20 in Table format.
- Attachment 5: included certificate of analysis of stock standards to list concentration of each element in the mix.

#### Attachment 1

## Working Standard Concentration for ICP Elements in ug/L

Element	CAL1	CAL2	CAL3/ICV/ CCV	Cal4	ICSA	ICSAB
Al	200	25000	125000	250000	500000	500000
Sb	10	200	1000	2000	n/a	100
As	5	500	2500	5000	n/a	100
Ва	200	2000	10000	20000	n/a	100
Ве	2	200	1000	2000	n/a	100
В	50	200	1000	2000	n/a	100
Cd	4	250	1250	2500	n/a	100
Са	5000	25000	125000	250000	500000	500000
Cr	10	1000	5000	10000	n/a	100
Со	50	500	2500	5000	n/a	100
Cu	25	2500	12500	25000	n/a	100
Fe	150	20000	100000	200000	200000	200000
Pb	5	1500	7500	15000	n/a	100
Mn	15	1000	5000	10000	n/a	100
Mg	5000	25000	125000	250000	500000	500000
Мо	20	500	2500	5000	n/a	100
Ni	40	500	2500	5000	n/a	100
К	5000	10000	50000	100000	n/a	10000
Se	5	500	2500	5000	n/a	100
Ag	10	250	1250	2500	n/a	100
Na	5000	25000	125000	250000	n/a	10000
Sr	20	1000	5000	10000	n/a	100
TI	10	500	2500	5000	n/a	100
Sn	50	200	1000	2000	n/a	100
Ti	20	2000	10000	20000	n/a	100
V	50	500	2500	5000	n/a	100
Zn	30	500	2500	5000	n/a	100

## Attachment 2

		trix Spike ration in Solutior	<u>1 (ug/L)</u>
ELEMENT	<u>Aqueous</u> (Non-TCLP)	<u>TCLP</u>	Soil
Aluminum	2000	5000	4000
Antimony	500	1000	1000
Arsenic	2000	5000	4000
Barium	2000	10000	4000
Beryllium	50	1000	100
Cadmium	50	1000	100
Calcium	20000	20000	40000
Chromium	200	5000	400
Cobalt	500	1000	1000
Copper	250	1000	500
Iron	1000	1000	2000
Lead	500	5000	1000
Manganese	500	1000	1000
Magnesium	20000	20000	40000
Nickel	500	1000	1000
Potassium	20000	20000	40000
Selenium	2000	1000	4000
Silver	50	500	100
Sodium	20000	20000	40000
Thallium	2000	1000	4000
Vanadium	500	500	1000
Zinc	500	1000	1000
Boron	500	1000	1000
Molybdenum	500	1000	1000
Tin	500	1000	1000
Titanium	500	1000	1000
Strontium	500	1000	1000

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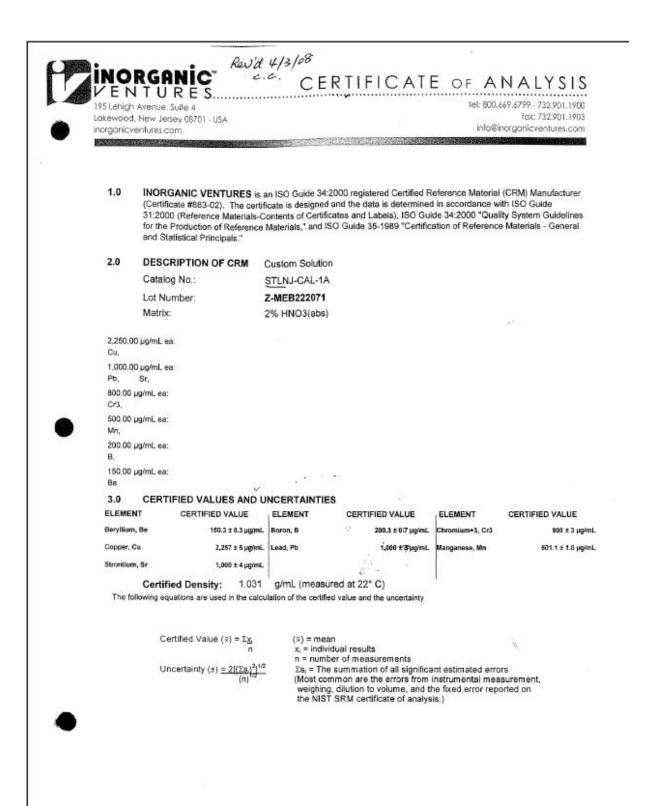
#### Attachment 3



**Company Confidential & Proprietary** 

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	anue, Suite 4 w Jarsey 08701 - USA ures.com				669.6799 - 732.901.190 fox: 732.901.190 inorganicventures.com
8 (19) (A) (19)					
(Ce 31: for	DRGANIC VENTURES artificate #863-02). The ca 2000 (Reference Materials the Production of Reference 1 Statistical Principels.*	rtificate is designed and Contents of Certificates	the data is determined in and Labels), ISO Guide Guide 35-1989 "Certifical	n accordance with ISO 34:2000 "Quality Sys ion of Reference Mate	i Guide tem Guidelines rials - General
2.0 DE	SCRIPTION OF CRM	Stock Solution	Dut-	Rec 10-0	Ŷ
- SIS 1973	talog No.:	CLPP-CAL-3	0.012	FCC	
	t Number:	A2-MEB194141	21	ve/ven	r
252	strix;	5% HNO3(abs)	1901 B.	li li	
1.00	0.00 µg/mL each:				
1,00 As,	Pb, Se, Ti,				
· 500. Cd	00 µg/mL each:				
3.0 CI	ERTIFIED VALUES AN	D UNCERTAINTIES			
ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT CEI	RTIFIED VALUE
Arsenic, As	1,002 ± 3 µg	tesL Cadmium, Cd	499.9 ± 1.2 µg/mL	Land, Pb	1,903 ± 3 µg/ml.
Selenium, Se	1,050 ± 2 µg	mL Thallium, 11	1,000 ± 3 µg/ml.		
The Certifie	ertified Density: 1.0 of Value is based upon the mo value and the uncertainty:		analyze this CRM. The folk	wing equations are used	in the calcutation of
	Certified Value (%) = Σg	x, ≠ individua			
	Uncertainty (±) <u>= 2[(Σs)</u> (n)	<sup>2</sup> 1 <sup>1/2</sup> Σε <sub>i</sub> = The su (Most comm weighing, d	of measurements immation of all significa- on are the errors from i llution to volume, and th RM certificate of analysi	nstrumental measure e fixed error reported	ment, on
4	0 TRACEBILITY TO N	IST AND VALUES O	BTAINED BY INDEP	ENDENT METHOD	S
0	Property of the result of a mea international standards, throu stimition 6.10)	igh an unbroken chain of C	omparisons all having stated	d uncertainties." (ISO VIA	I, 2nd ed., 1993,
te	This product is Traceable to M king into account the SRM un IST SRMs are available, the M	certainty error and the mea	surement, weighing and vo	iume dilution errors. In ra	are cases where no



	N T U ph. Avenue,	RES	413/08 CEI	RTIFICAT		ANALYSIS
		sey 08701 - USA			1048	fax: 732.901.1983
	oventures.c					s@inorganicventures.com
and a second second			Man Market Dirt Jok 2715		CONTRACTO DI LE CONTRA	
1.0	(Certifi 31:200 for the	icate #663-02). The cert 00 (Reference Materials-	ificate is designed a Contents of Certification of Certification (Contents of Certification (Contents of Certification (Certification (Certi	1000 registered Certified F and the data is determined ates and Labels), ISO Gu O Guide 35-1989 "Certific	d in accordance ide 34:2000 "Q	s with ISO Guide uality System Guidelines
102.027						
2.0		RIPTION OF CRM	Custom Solution			
		og No.:	STLNJ-CAL-1B			
	13334033	umber:	Z-MEB222072			
	Matrix		tr. HF,	2% HNO3(abs)		
2,000 Ti,	1.00 µg/mi_ e	9:				
	0 µg/mL ea:					
Mo,	20000000					
200.0 Sb.	0 µg/mL ea: Sn					
3.0		IFIED VALUES AND I	INCERTAINTIES	1		
		The Predeo And				
-ELEM	ENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALUE
8 (S.23)		CERTIFIED VALUE 199.2 ± 0.6 µg/ml	ELEMENT Molybdenum, Mo	CERTIFIED VALUE 499.4 ± 1.3 µg/mL	ELEMENT	CERTIFIED VALUE
ELEM	my, Sb	199.2 ± 0.6 µg/ml	Molybdenum, Mo			
Antimo	my, Sb m, Ti	199.2 ± 0.6 µg/ml 2,004 ± 4 µg/ml	Molybdenum, Mo	499.4 ± 1.3 µg/mL		
ELEM Antimo Titaniu	my, Sb m, Ti Certifi	199.2 ± 0.6 µg/mL 2,004 ± 4 µg/mL led Density: 1.021	g/mL (measure	499.4 ± 1.3 µg/mL ed at 22° C)		
ELEM Antimo Titaniu	my, Sb m, Ti Certifi	199.2 ± 0.6 µg/ml 2,004 ± 4 µg/ml	g/mL (measure	499.4 ± 1.3 µg/mL ed at 22° C)		
ELEM Antimo Titaniu	my, Sb m, Ti Certifi following eqi	199.2 ± 0.4 µg/ml 2,604 ± 4 µg/ml ied Density: 1.021 uations are used in the calc	g/mL (measure	499.4 ± 1.3 µg/mL ed at 22° C)		
ELEM Antimo Titaniu	my, Sb m, Ti Certifi following eqi	199.2 ± 0.6 µg/mL 2,004 ± 4 µg/mL led Density: 1.021	g/mL (measure ulation of the certified (x) = mean	499.4 ± 1.3 µg/mL ed at 22° C) I value and the uncertainty		
ELEM Antimo Titaniu	my, Sb m, Ti following eq Ce	199.2 ± 0.6 µg/mL 2,004 ± 4 µg/mL led Density: 1.024 uations are used in the calo entified Value $(z) = \sum_{\underline{x}_i}$ n	(x) = mean x, = individ n = number	499.4 ± 1.3 µg/mL ed at 22° C) Value and the uncertainty ual results	Tin, Sa	196.4 ± 0.6 µgh
ELEM Antimo Titaniu	my, Sb m, Ti following eq Ce	199.2 ± 0.4 µg/ml 2,604 ± 4 µg/ml ied Density: 1.021 uations are used in the calc	(x) = mean x, = individ x, = individ x, = individ	499.4 ± 1.3 µg/mL ed at 22° C) I value and the uncertainty ual results in of measurements summation of all significa	Tin, Se	196.4 ± 0.6 µg4
-ELEM Antimo Titanio	my, Sb m, Ti following eq Ce	199.2 ± 0.6 µg/mL 2,004 ± 4 µg/mL led Density: 1.024 uations are used in the calo entified Value $(z) = \sum_{\underline{x}_i}$ n	Molybdenum, Mo g/mL (measure ulation of the certified (x) = mean x <sub>i</sub> = individi n = numbe Σs <sub>i</sub> = The s (Most comi weighing,	499.4 ± 1.3 µg/mL ed at 22° C) value and the uncertainty ual results r of measurements summation of all significa mon are the errors from i dilution to volume, and th	Tin, Sa nt estimated e instrumental m te fixed error m	196.4 ± 0.6 µgh ITOITS 805urement.
ELEM Antimo Titaniu	my, Sb m, Ti following eq Ce	199.2 ± 0.6 µg/mL 2,004 ± 4 µg/mL led Density: 1.024 uations are used in the calo entified Value $(z) = \sum_{\underline{x}_i}$ n	Molybdenum, Mo g/mL (measure ulation of the certified (x) = mean x <sub>i</sub> = individi n = numbe Σs <sub>i</sub> = The s (Most comi weighing,	499.4 ± 1.3 µg/mL ed at 22° C) value and the uncertainty ual results of measurements summation of all significa mon are the errors from i	Tin, Sa nt estimated e instrumental m te fixed error m	1964±04 µgh rrors easurement.
ELEM Antimo Titaniu	my, Sb M, Ti following eq Ce Un	199.2 ± 0.5 µg/ml 2,604 ± 4 µg/ml led Density: 1.024 uations are used in the calc entified Value $(x) = \sum_{\underline{x}}$ n identified Value $(x) = \sum_{\underline{x}}$ n identified Value $(x) = \sum_{\underline{x}}$ (n)	Molybdenum, Mo g/mL (measure ulation of the certified (x) = mean x, = individ n = numbe Es; = The (Most com weighing, the NIST (	499.4 ± 1.3 µg/mL ed at 22° C) value and the uncertainty ual results r of measurements summation of all significa mon are the errors from i dilution to volume, and th	Tin, Se nt estimated e Instrumental m le fixed error m is.)	196,4 ± 0.6 µgH frors easurement, sported on
ELEM Antimo Titaniu	my, Sb m, Ti following eq Ce Un 4.0 Ti	199.2 ± 0.6 µg/mL 2,604 ± 4 µg/mL led Density: 1.021 uations are used in the calc entified Value $(x) = \sum_{\underline{x}_1} \frac{1}{n}$ incertainty $(\pm) = 2[(\Sigma_{\underline{x}_1})^2]^{11} \frac{1}{(n)^{1/2}}$ RACEBILITY TO NIST	Molybdenum, Mo g/mL (measure ulation of the certified (x) = mean x <sub>i</sub> = individi n = numbe Ss <sub>i</sub> = The s (Most com weighing, the NIST s	499.4 ± 1.3 µg/mL ed at 22° C) value and the uncertainty ual results of measurements summation of all significa mon are the errors from i diution to volume, and th SRM certificate of analysi	Tin, Sa Int estimated e Instrumental m te fixed error m is.) ENDENT ME	196.4 ±0.6 µg/r easurement, sported on
ELEM Antimo Titaniu	my, Sb m, Ti following equ Ce Un 4.0 Ti · "Proper	199.2 ± 0.6 µg/ml 2,604 ± 4 µg/ml led Density: 1.024 uations are used in the calc entified Value $(x) = \sum_{\underline{X}_1} \frac{1}{n}$ incertainty $(\pm) = 2[(\Sigma_0)^2]^{1/2}$ RACEBILITY TO NIS <sup>-</sup> thy of the result of a measure etional standards, through a	Molybdenum, Mo g/mL (measure ulation of the certified (x) = mean x, = individ n = numbe Σs <sub>i</sub> = The s (Most com weighing, the NIST ( TAND VALUES (	499.4 ± 1.3 µg/mL ed at 22° C) value and the uncertainty ual results of measurements summation of all significa mon are the errors from i diution to volume, and th SRM certificate of analysi	Tin, Se nt estimated e instrumental m re fixed error re is .) ENDENT MET e related to state	196.4 ± 0.6 µght easurement, sported on FHODS d references, usually pations

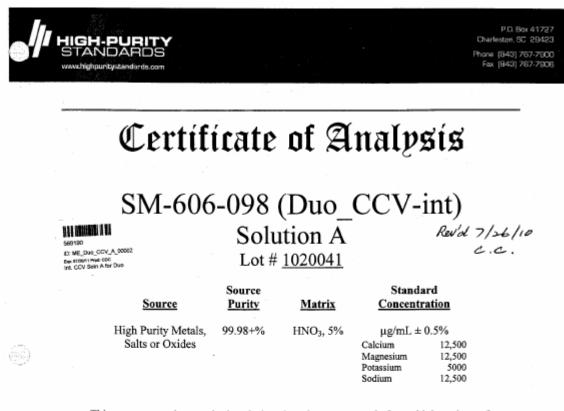


"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10)

 This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

	nicventures.c					
						info@inorganicventu
1.0	Competer manufact	nce of Reference I urer. Our manufa Requirements fo	Material Producers cturing laboratory i	"General Requireme " and ISO 9001:2000 is accredited to ISO/I ise of Testing and (	registered EC 17025	
2.0	DESCRIP	TION OF CRM	Custom Solution			
	Catalog N	lo.:	TANJ-STD-3			
	Lot Numb	er:	D2-MEB325088			
	Matrix:		5% HNO3(v/v)			
500.00	unimi ess					
500.00 Ca,	µg/mLea: K, Mg,	Na,				
20.00 µį Al,	gímL ea: Ba,					
	g/mL ea:					
Fe, 5.00 µg/	mi ea-					
Co,	V,					
4.00 μg/ Ni,	mL ea:					
3.00 µg/	mL ea:					
Zn,						
2.50 µg/ Cu,	mL ea:					
2.00 µg/	mL ea:					
Sr,						
1.50 µg/ Мо,	mL ea:					
1.00 µg/	mL ea:					
Ag.	Cr3, Ti,					
0.50 µg/ As,	mLea: Pb, Se,					
0.40 µg/ Cd,	mL ea:					
0.20 μg/i Be	mL ea:					
3.0	CERTIFIE	D VALUES AND	UNCERTAINTIES			
		ERTIFIED VALUE	ELEMENT	CERTIFIED VALUE	ELEMENT	CERTIFIED VALU
ELEMEN			Annual A	0.4000	Dealer Dealer	
Aluminum	, Al	20.02 ± 0.04 µg/mL	Arsenic, As	0.4980 ± 0.0023 µg/mL	Barium, Ba	19.97 ± 0.0
		20.02 ± 0.04 µg/mL 0.2000 ± 0.0005 µg/mL		0.4980 ± 0.0023 µg/mL	Barium, Ba Calcium, Ca	19.97 ± 0.0 499.6 ± 1.

Chris	echnology	VA 24073 - USA		ERTIFICA	tel: 8	00.669.6799 - 540.58: fox: 540.58: x@inarganicventure:
1.0	Comp manuf "Gene	etence of Reference facturer. Our manufa	Material Produce cturing laborator	34 "General Requiremer ers" and ISO 9001:2000 r ry is accredited to ISO/IE ence of Testing and C	egistered	
2.0	DESC	RIPTION OF CRM	Custom Solutio	on		
	Catalo	g No.:	TANJ-STD-4			
	Lot Nu	imber:	D2-MEB32508	37		
	Matrix:		tr. HF,	5% HNO3(v/v)		
ELEMI Antimo Tin, Sn		CERTIFIED VALUE 1.004 ± 0.007 µg/m 5.009 ± 0.013 µg/m		CERTIFIED VALUE 5.019 ± 0.017 µg/mL 1.998 ± 0.009 µg/mL	ELEMENT Molybdenum, Mo	CERTIFIED VALUE 1.999 ± 0.009 (
The f unce	ollowing equ	ed Density: 1.024 Jations are used in the cali- ressed at approximately the	culation of the certifi	ured at 20 ± 1°C) ed value and the uncertainty. F evel using a coverage factor of	Reported uncertaintie k = 2.	s represent expandad
		rtified Value (z) = $\sum_{\underline{N}} \frac{1}{n}$ certainty (±) = $2[(\sum_{n})^{2}]$ (n) <sup>1/2</sup>	n = numt Σs, = The (Most co weighing	an oer of measurements e summation of all significa mmon are the errors from i 1, diution to volume, and th f SRM certificate of analysi	nstrumental meas e fixed error repor	urement
4.0	TRACE	BILITY TO NIST AN	D VALUES OB	FAINED BY INDEPENDE	NT METHODS	erences, usually natic



This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

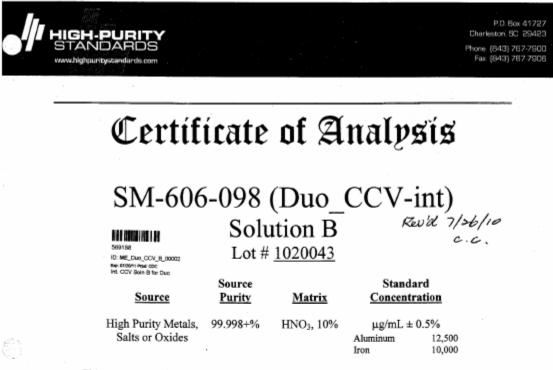
Theodor C Raine

Theodore C. Rains, Ph.D. President

Exp Date: JUL 2 0 2011 MSDS ATTACHED

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SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 31 of 47



This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

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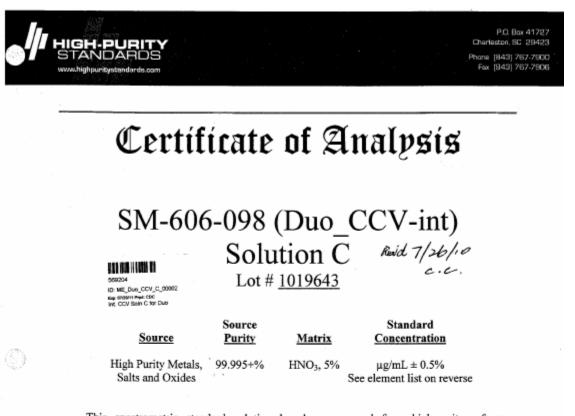
In C Raine

Exp Date: JUL 2 0 2011 MSDS ATTACHED

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Theodore C. Rains, Ph.D. President

SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 32 of 47



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Theodor C Raine

Theodore C. Rains, Ph.D. President

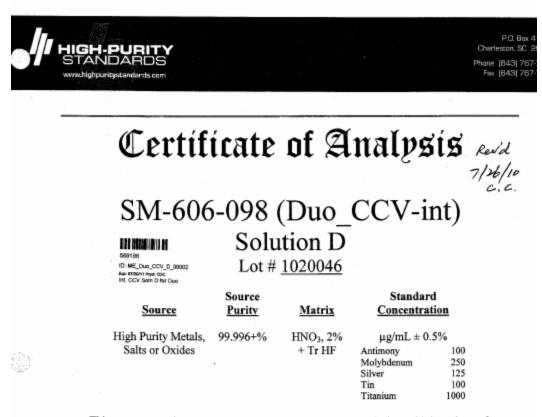
Exp Date: JUL 2 0 2011 MSDS ATTACHED

SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 33 of 47

## SM-606-098 (Duo\_CCV-int) Solution C Element List (µg/mL)

Arsenic	250
Barium	1000
Beryllium	100
Boron	100
Cadmium	125
Chromium	500
Cobalt	250
Copper	1250
Lead	750
Manganese	500
Nickel	250
Selenium	250
Strontium	500
Thallium	250
Vanadium	250
Zinc	250

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This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodor C Raine

Theodore C. Rains, Ph.D. President

Exp Date: JUL 2 0 2011 MSDS ATTACHED

SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 35 of 47

	: 800.878.7654 : 707.545.7901	Europe P.O. Box 2704 P. + 31 20 438 05 97 1000 CS Amsterdam P: + 31 20 420 28 36 The Netherlands P. + 31 20 420 28 36 The Netherlands Www.cplinternationd.com www.cplinternationd.com WWW.cplinternationd.com
Expiry: 21-Sep-12		DE
	<u>Certifica</u>	ite of Analysis
Part Number: Lot Number: Shelf Life:	4400-100419DD0 <sup>.</sup> 11C157 18 Months	O3/22/11MP)
TestAmerica/Edis Custom Standard 5% HNO3		COST ZZINI MP
Concentrations in ug/	/mL ± 0.5%	
Fe         1000           Se         2000           TL         2000           Be         50           Cd         50           Cr         200           Co         500           Cu         250           Pb         500           Ag         50           Sr         500           V         500	Zn 500 Mn 500 Ni 500	
required) and 18-m significant figures an Starting materials w solution concentratio	egaohm de-ionized w d diluted in volumetric ere analyzed at 1000 ons were certified instr	ng high-purity starting materials, high-purity acid (if ater. The starting materials were weighed to five glassware calibrated to five significant figures. Ig/mL by ICP-MS for trace impurities. The standard umentally against the National Institute of Standards approved second source and/or gravimetrically.
Accuracy and stabili stated shelf life from	ty are guaranteed to w the date of shipment.	within plus or minus 0.5% of the certified value for the The solution should be kept tightly capped and stored tached MSDS for proper handling information.
	nments please call 1-8 at www.cpiinternationa	00-878-7654 in the USA, +31 20 638 05 97 in Europe

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USA 5580 Skylane Boulevard P: 707,525.5788 Santa Rosa, CA 95403 P: 800,878,7654 P: 707,545,7901



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 Europe

 P.O. Box 2704
 P: +31 20 638 05 97

 1000 CS Amsterdiam
 P: +31 20 420 28 36

 The Netherlands
 P: +31 20 420 28 36

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: Lot Number: Shelf Life: 4400-100419DD01 11C157 18 Months Solution B

TestAmerica/Edison Custom Standard 10% HNO3

Dute Reald. U3/122/11 MP

Concentrations in ug/mL ± 0.5%

Al	2000
Mg	20000
ĸ	20000
Ca	20000
Na	20000

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megaohm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cplinternational.com

SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 37 of 47

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Expiry: 21	-Sep-1	2		. /
		<u>Certificate</u>	<u>of Analysis</u>	
Part Numb Lot Numbe Shelf Life:		4400-100419DD01 11C157 18 Months	Solution C	
			. DAte	Locly
TestAmeric Custom Sta 5% HNO3 +	ndard	1	03/11	Locky -/11 Mie
Concentration	s in ug	/mL ± 0.5%		
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olution conce	ntratio	ns were certified instrumentally	against the National Institute of Standards econd source and/or gravimetrically.	
tated shelf life	e from	the date of shipment. The solut	or minus 0.5% of the certified value for the on should be kept tightly capped and stored SDS for proper handling information.	
or questions	or com b-site a	ments please call 1-800-878-76 t www.cpiinternational.com.	54 in the USA, +31 20 638 05 97 in Europe	

SOP No. ED-MT-004, Rev. 7 Effective Date: 04/25/2011 Page No.: 38 of 47

USA	
i580 Skylane Boulevard ianta Rosa, CA 95403	P: 707.525.5788 P: 800.878.7654
	F: 707.545.7901



Europe P.O. 8ax 2704 P: +31 20 638 05 97 1000 CS Amsterdam F: +31 20 420 28 36 The Netherlands

Adranoni Analytical, Seniconductor and Life Science Solution www.cpiinternational.com www.colilog.com

Expiry: 21-Sep-12

Certificate of Analysis

Part Number: Lot Number: Shelf Life: 4400-100419DD01 11C157 18 Months Solution D

OS/22/11 Mp

TestAmerica/Edison Custom Standard 2% HNO3

Concentrations in ug/mL ± 0.5%

As	2000
Ba	2000
R	500

This standard solution was prepared using high-purity starting materials, high-purity acid (if required) and 18-megachm de-ionized water. The starting materials were weighed to five significant figures and diluted in volumetric glassware calibrated to five significant figures.

Starting materials were analyzed at 1000µg/mL by ICP-MS for trace impurities. The standard solution concentrations were certified instrumentally against the National Institute of Standards and Technology's SRM 3100 series, NIST approved second source and/or gravimetrically.

Accuracy and stability are guaranteed to within plus or minus 0.5% of the certified value for the stated shelf life from the date of shipment. The solution should be kept tightly capped and stored under normal laboratory conditions. See attached MSDS for proper handling information.

For questions or comments please call 1-800-878-7654 in the USA, +31 20 638 05 97 in Europe or visit our web-site at www.cpiinternational.geg.

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The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodor C Raine

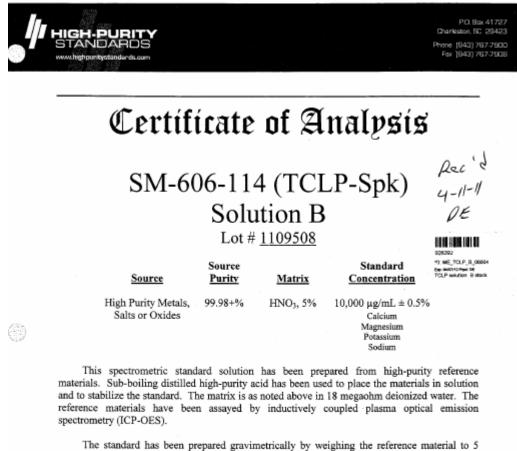
APR 0 7 2012

Theodore C. Rains, Ph.D.

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### SM-606-114 (TCLP-Spk) Solution A Element List (µg/mL)

Barium 5000 Beryllium 500 Cadmium 500 Cobalt 500 Copper 500 Iron 500 Manganese 500 Nickel 500 500 Selenium Strontium 500 Thallium 500 Zinc 500



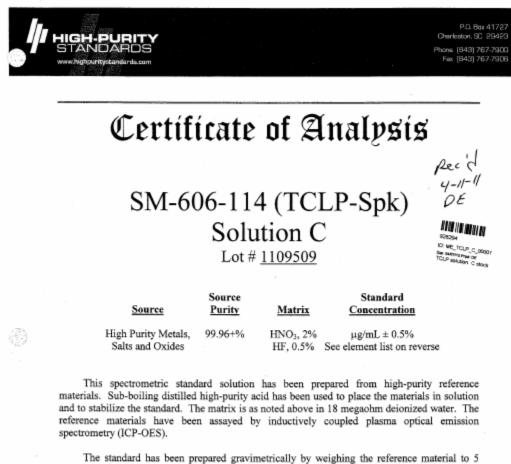
The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

APR 0 7 2012 Exp Date: MSDS ATTACHED

Theodor C Raine Theodore C. Rains, Ph.D. President

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The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

In C Raina

Theodore C. Rains, Ph.D. President

Exp Date: APR 0 7 2012

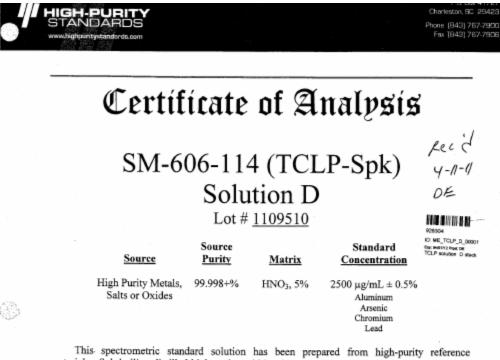
64

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# SM-606-114 (TCLP-Spk) Solution C Element List

 	m.	

Anitmony	500
Boron	500
Molybdenum	500
Silver	250
Tin	500
Titanium	500
Vanadium	250



This spectrometric standard solution has been prepared from high-purity reference materials. Sub-boiling distilled high-purity acid has been used to place the materials in solution and to stabilize the standard. The matrix is as noted above in 18 megaohm deionized water. The reference materials have been assayed by inductively coupled plasma optical emission spectrometry (ICP-OES).

The standard has been prepared gravimetrically by weighing the reference material to 5 significant figures. Volumetric glassware has been calibrated gravimetrically to 5 significant figures. The standard concentration has been verified by ICP-OES against an independent source which is directly traceable to National Institute of Standards and Technology, Standard Reference Material No. 3100 series.

This standard is valid for one year from the shipping date provided the solution is kept tightly capped and stored under normal laboratory conditions.

Theodor C Paine

Exp Date: APR 0 7 2012 MSDS ATTACHED

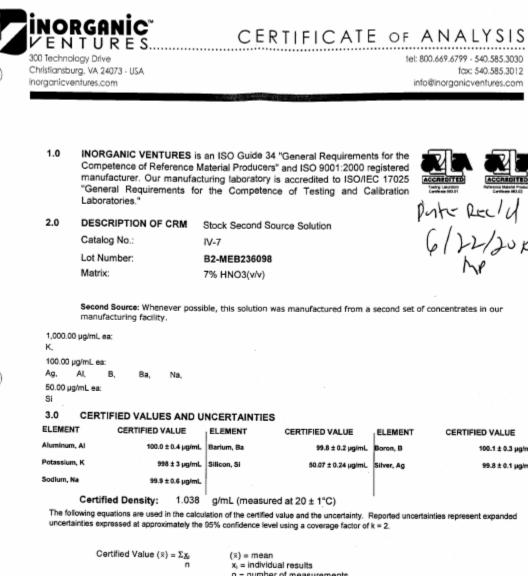
Theodore C. Rains, Ph.D. President

		RGANIC TURES logy Drive urg, VA 24073 - USA entures.com		CER	RTIFICATE	tel: 800.	.669.6799 - 540.585.3 fax: 540.585.3 linorganicventures.c
Competence of Reference Material Producers" and ISO 9001 registered manufacture. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories." <b>2.0 DESCRIPTION OF CRM</b> Stock Solution Catalog No.: CLPP-ICS-A Lot Number: D2-MEB324151MCA Matrix: 2% HNO3(v/v) <b>5.000.00 µg/mL ea:</b> A, Ca, Mg, <b>2.000.00 µg/mL ea:</b> Fe <b>3.0 CERTIFIED VALUE</b> ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE Calcian, Ca 5,000 ± 20 µg/mL from, Fe 2,000 Magnesium, Mg 5,000 ± 18 µg/mL Calcian, Ca 5,000 ± 20 µg/mL from, Fe 2,000 The following equations are used in the calculation of the cartified value and the uncertainty. Reported uncertainties represent ex uncertainties expressed at approximately the BS% confidence level using a coverage factor of k = 2. Certified Value ( $\hat{x} = \sum_{k}$ , $(\hat{x}) = mean$ $n$ $x_{k} = individual results$ n = number of measurements Uncertainty ( $t) = 2[(\sum_{k})^{2}]^{4}$ $\sum_{k}$ The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the					1		
Competence of Reference Material Producers" and ISO 9001 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories." <b>2.0 DESCRIPTION OF CRM</b> Stock Solution Catalog No.: CLPP-ICS-A Lot Number: D2-MEB324151MCA Matrix: 2% HNO3(v/v) <b>5.000.00 µg/mL es:</b> A, Ca, Mg, 2,000.00 µg/mL ea: Fe <b>3.0 CERTIFIED VALUE</b> ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE Calcian, Ca 5,000 ± 20 µg/mL (ron, Fe 2,000 Magnesium, Mg 5,000 ± 18 µg/mL Calciam, Ca 5,000 ± 20 µg/mL (ron, Fe 2,000 Magnesium, Mg 5,000 ± 18 µg/mL Calciam, Ca 5,000 ± 20 µg/mL (ron, Fe 2,000 The following equations are used in the calculation of the cartified value and the uncertainty. Reported uncertainties represent ex uncontainties expressed at approximately the BS% confidence level using a coverage factor of k = 2. Certified Value ( $\hat{x} = \sum_{i=1}^{N} (\hat{x}) = mean$ $n$ $x_i= individual results$ n = number of measurements $Uncertainty (t) = 2[(\Sigma_S_1)^2]^N$ $\Sigma_S = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the$							
Catalog No.:CLPP-ICS-ALot Number:D2-MEB324151MCAMatrix:2% HNO3(v/v)5,000.00 µg/mL ea:Ai, Ca, Mg,2,000.00 µg/mL ea:Fe3.0 CERTIFIED VALUES AND UNCERTAINTIESELEMENTCERTIFIED VALUEAluminum, Ai5,000 ± 18 µg/mLCalcium, Ca5,000 ± 20 µg/mLIron, Fe2,000Magnesium, Mg5,000 ± 18 µg/mLCalcium, Ca5,000 ± 20 µg/mLIron, Fe2,000Magnesium, Mg5,000 ± 18 µg/mLCatrified Density:1.085g/mL (measured at 20 ± 1° C)The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expunce the server seed at approximately the 95% confidence level using a coverage factor of k = 2.Certified Value ( $\overline{X}$ ) = $\underline{\Sigma} \underline{X}_i$ ( $\overline{X}$ ) = mean n $X_i$ = individual results n = number of measurements $\Sigma_{5i}$ = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	1.0	Competence manufacturer. "General Rec	of Reference Our manufac quirements fo	Material Producturing laboratory	cers" and ISO 9001 re is accredited to ISO/IE	c 17025	
Lot Number:D2-MEB324151MCAMatrix:2% HNO3(v/v)5,000.00 µg/mL ea:A,Ca,A,Ca,Matrix:2% HNO3(v/v)5,000.00 µg/mL ea:Fe <b>3.0 CERTIFIED VALUES AND UNCERTAINTIES</b> ELEMENTCERTIFIED VALUEAluminum, AI5,000 ± 18 µg/mLCalcium, Ca5,000 ± 20 µg/mLImagenesium, Mg5,000 ± 16 µg/mLCertified Density:1.085Imagenesium, Mg5,000 ± 16 µg/mLCertified Density:1.085g/mL (measured at 20 ± 1° C)The following equations are used in the calculation of the certified value and the uncertainties represent explored uncertainties represent explored uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.Certified Value ( $\overline{X}$ ) = $\underline{\Sigma} \underline{X}_1$ ( $\overline{X}$ ) = mean n $X_1$ = individual results n = number of measurements $\Sigma_5$ = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	2.0	DESCRIPTIO	N OF CRM	Stock Solution			
Matrix:2% HNO3(v/v)\$,000.00 µg/mL ea: Al, Ca, Mg, 2,000.00 µg/mL ea: Fe <b>3.0</b> CERTIFIED VALUES AND UNCERTAINTIESELEMENTCERTIFIED VALUE Cartified VALUE Name Magnesium, MgELEMENTCERTIFIED VALUE Calcium, CaELEMENTCERTIFIED VALUE Calcium, CaMagnesium, Mg5,000 ± 18 µg/mL Cortified Density:1.085 1.085g/mL (measured at 20 ± 1° C)ELEMENTCERTIFIED value Cortified Density:ELEMENTCERTIFIED value Cartified value and the uncertainty. Reported uncertainties represent expunction uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.Certified Value nnx = individual results n = number of measurements Sare The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the		Catalog No.:		CLPP-ICS-A			
5,000.00 µg/mL ea: Al, Ca, Mg, 2,000.00 µg/mL ea: Fe 3.0 CERTIFIED VALUES AND UNCERTAINTIES ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE Aluminum, Al 5,000 ± 18 µg/mL Calcium, Ca 5,000 ± 20 µg/mL fron, Fe 2,000 Magnesium, Mg 5,000 ± 16 µg/mL g/mL (measured at 20 ± 1° C) The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expluit uncertainties expressed at approximately the 95% confidence level using a covarage factor of k = 2. Certified Value $(\vec{x}) = \sum_{i=1}^{N} (\vec{x}) = mean$ n $x_i = individual results$ n = number of measurements Uncertainty $(\pm) = 2[(\sum_{i=1}^{N})^2]^{\frac{N}{2}}$ $\sum_{i=1}^{N}$ The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the		Lot Number:		D2-MEB324151	IMCA		
Ai, Ca, Mg, 2,000.00 µg/mL ea: Fe <b>3.0 CERTIFIED VALUES AND UNCERTAINTIES</b> <b>ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE ELEMENT CERTIFIED VALUE</b> Aluminum, AI 5,000 ± 18 µg/mL Calcium, Ca 5,000 ± 20 µg/mL from, Fe 2,000 Magnesium, Mg 5,000 ± 16 µg/mL g/mL (measured at 20 ± 1° C) The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expuncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2. Certified Value $(\vec{x}) = \sum_{i=1}^{N} (\vec{x}_i) = mean$ n = number of measurements Uncertainty $(\pm) = 2[(\sum_{i=1}^{N})^2]^{\frac{N}{2}}$ $\sum_{i=1}^{N}$ In the summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the		Matrix:		2% HNO3(v/v)			
ELEMENT       CERTIFIED VALUE       ELEMENT       CERTIFIED VALUE       ELEMENT       CERTIFIED VALUE         Aluminum, Al       5,000 ± 18 µg/mL       Calcium, Ca       5,000 ± 20 µg/mL       Iron, Fe       2,000         Magnesium, Mg       5,000 ± 16 µg/mL       Calcium, Ca       5,000 ± 20 µg/mL       Iron, Fe       2,000         Certified Density:       1.085       g/mL (measured at 20 ± 1° C)       The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expuncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.       Certified Value ( $\vec{X}$ ) = $\vec{\Sigma}$ x ( $\vec{x}$ ) = mean       ( $\vec{x}$ ) = mean       n       x <sub>1</sub> = individual results       n = number of measurements         Uncertainty ( $\pm$ ) = $2[(\Sigma s_1)^2]^{\frac{N}{2}}$ $\vec{\Sigma}$ si = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the							
Aluminum, Al       5,000 ± 18 µg/mL       Calcium, Ca       5,000 ± 20 µg/mL       Iron, Fe       2,000         Magnesium, Mg       5,000 ± 16 µg/mL       Calcium, Ca       5,000 ± 20 µg/mL       Iron, Fe       2,000         Certified Density:       1.085       g/mL (measured at 20 ± 1° C)       Iron, Fe       2,000         The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent explored uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.       Certified Value ( $\vec{x}$ ) = $\vec{\Sigma} \times I$ , ( $\vec{x}$ ) = mean n = number of measurements n = number of measurements       Iron, Fe       2,000         Uncertainty ( $\pm$ ) = $2[(\Sigma s_1)^2]^{\frac{N}{2}}$ $\vec{\Sigma}$ spin the summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	Al, 2,000.0	Ca, Mg,					
Magnesium, Mg       5,000 ± 16 µg/mL         Certified Density:       1.085       g/mL (measured at 20 ± 1° C)         The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent explored tables expressed at approximately the 95% confidence level using a coverage factor of k = 2.         Certified Value ( $\vec{x}$ ) = $\sum_{n} x_i$ ( $\vec{x}$ ) = mean       ( $\vec{x}$ ) = mean         n $x_i$ = individual results         n       n = number of measurements         Uncertainty ( $\pm$ ) = $2[(\Sigma s_i)^2]^{3/3}$ $\Sigma s_i$ = The summation of all significant estimated errors         (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	AI, 2,000.0 Fe <b>3.0</b>	Ca, Mg, 30 µg/mL ea: CERTIFIED V	ALUES AND	UNCERTAINTIE	s		*
Certified Density:       1.085       g/mL (measured at 20 ± 1° C)         The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent explored the expressed at approximately the 95% confidence level using a coverage factor of k = 2.         Certified Value ( $\vec{x}$ ) = $\sum_{k} x_{i}$ ( $\vec{x}$ ) = mean         n $x_{i}$ = individual results         n $n$ in the comber of measurements         Uncertainty (±) = $2[(\sum_{i})^{2}]^{\frac{N}{2}}$ $\sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{i=1}^{N} \sum_{j=1}^{N} \sum_{j=1}^{N$	AI, 2,000.0 Fe <b>3.0</b>	Ca, Mg, 30 µg/mL ea: CERTIFIED V				ELEMENT	CERTIFIED VAL
The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expressed at approximately the 95% confidence level using a coverage factor of k = 2. Certified Value ( $\bar{x}$ ) = $\sum x_i$ ( $\bar{x}$ ) = mean $x_i$ = individual results $n = number of measurements$ Uncertainty ( $\pm$ ) = $2[(\Sigma s_i)^2]^{\frac{N}{2}}$ ( $\bar{x}$ )s;= The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	AI, 2,000.0 Fe <b>3.0</b> ELEME	Ca, Mg, 00 µg/mL ea: CERTIFIED V NT CERT	IFIED VALUE	ELEMENT	CERTIFIED VALUE		CERTIFIED VAL 2,000 ± 6
uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2. Certified Value $(\bar{x}) = \underbrace{\sum x_i}_{n}$ $(\bar{x}) = mean$ $x_i = individual results$ n = number of measurements Uncertainty $(\pm) = \frac{2[(\sum s_i)^2]^{\times}}{(n)^{\times}}$ $\sum_{x_i = The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the$	Al, 2,000.0 Fe <b>3.0</b> ELEME	Ca, Mg, 00 µg/mLea: CERTIFIED V NT CERT m, Al	fified VALUE 5,000 ± 18 µg/m	ELEMENT L Calcium, Ca	CERTIFIED VALUE		
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$\begin{array}{ll} n & x_i = \text{ individual results} \\ n = \text{ number of measurements} \\ \text{Uncertainty } (\pm) = \frac{2[(\Sigma s_i)^2]^{\times}}{(n)^{\times}} & \sum_{i \neq i \neq i} \text{ The summation of all significant estimated errors} \\ (\text{Most common are the errors from instrumental measurement,} \\ \text{weighing, dilution to volume and the fixed error reported on the} \end{array}$	Al, 2,000.0 Fe <b>3.0</b> ELEME! Magnes! The fe	Ca, Mg, D0 µg/mL ea: CERTIFIED V NT CERT m, Al lum, Mg Certified Den ollowing equations a	11FIED VALUE 5,000 ± 18 µg/m 5,000 ± 16 µg/m nsity: 1.084 re used in the calo	ELEMENT Calcium, Ca L 5 g/mL (measu culation of the certifie	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R	Iron, Fe	2,000 ± 6
Uncertainty $(\pm) = 2[(\Sigma s_1)^2]^{\frac{1}{2}}$ (n) <sup>2</sup> n = number of measurements $\Sigma s_i =$ The summation of all significant estimated errors (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the	Al, 2,000.0 Fe 3.0 ELEME; Aluminu Magnesi The fc uncer	Ca, Mg, 20 µg/mL ea: CERTIFIED V NT CERT m, Al turn, Mg Certified Den oliowing equations at tainties expressed a	TIFIED VALUE 5,000 ± 18 µg/m 5,000 ± 16 µg/m nsity: 1.085 re used in the calo t approximately th	ELEMENT Calcium, Ca Goldan, Ca Calcium, Calcium, Calci	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R	Iron, Fe	2,040 ± 6
<ul> <li>(n) (Most common are the errors from instrumental measurement, weighing, dilution to volume and the fixed error reported on the</li> </ul>	Al, 2,000.0 Fe <b>3.0</b> ELEME: Aluminu Magnesi The fc uncer	Ca, Mg, 20 µg/mL ea: CERTIFIED V NT CERT m, Al turn, Mg Certified Den oliowing equations at tainties expressed a	TIFIED VALUE $5,000 \pm 18 \mu g/m$ $5,000 \pm 16 \mu g/m$ insity: 1.085 inter used in the calculated on	ELEMENT Calcium, Ca Gy/mL (measure culation of the certifie e 95% confidence lev ) = mean	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R vel using a coverage factor of k	Iron, Fe	2,000 ± 6
<ul> <li>weighing, dilution to volume and the fixed error reported on the</li> </ul>	Al, 2,000.0 Fe <b>3.0</b> ELEME Aluminu Magnesi The fc uncert	Ca, Mg, D0 µg/mL ea: CERTIFIED V NT CERT m, Al hum, Mg Certified Den blowing equations at tainties expressed at tainties expressed at	TIFIED VALUE 5,000 ± 18 µg/m 5,000 ± 16 µg/m nsity: 1.085 re used in the calk t approximately th $\frac{\sum x}{n}$ , $(\bar{x})$ n = n =	ELEMENT Calcium, Ca Calcium, Ca 5 g/mL (measu culation of the certifie te 95% confidence lew ) = mean individual results number of meas	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R vel using a coverage factor of k	ported uncertain	2,040 ± 6
NIST SRM certificate of analysis)	Al, 2,000.0 Fe <b>3.0</b> ELEME! Aluminu Magnes! The fe uncer	Ca, Mg, D0 µg/mL ea: CERTIFIED V NT CERT m, Al hum, Mg Certified Den blowing equations at tainties expressed at tainties expressed at	TIFIED VALUE 5,000 ± 18 µg/m 5,000 ± 16 µg/m nsity: 1.085 re used in the calk the used in the calk the used in the calk the used in the calk re use	ELEMENT Calcium, Ca Calcium, Ca S g/mL (measult culation of the certifier e 95% confidence lev ) = mean individual results : number of meas = The summation	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R vel using a coverage factor of k s surements n of all significant estimal	eported uncertain t = 2.	2,000 ± 6 nties represent expan
	Al, 2,000.0 Fe <b>3.0</b> ELEME! Aluminu Magnes! The fe uncer	Ca, Mg, D0 µg/mL ea: CERTIFIED V NT CERT m, Al hum, Mg Certified Den blowing equations at tainties expressed at tainties expressed at	TIFIED VALUE 5,000 ± 18 µg/m 5,000 ± 16 µg/m hsity: 1.085 re used in the call the tapproximately th $\frac{\sum x}{n}$ , $(\bar{x}, x)$ $\frac{x}{n}$ $\frac{x}{(\bar{x})^2}$ , $(\bar{x}, w)$ $(m)^2$	ELEMENT Calcium, Ca Calcium, Ca 5 g/mL (measu culation of the certifie 95% confidence lev ) = mean individual results number of measures = The summation ost common are to ighing, dilution to	CERTIFIED VALUE 5,000 ± 20 µg/mL red at 20 ± 1° C) d value and the uncertainty. R wel using a coverage factor of k surements n of all significant estimat the errors from instrumer volume and the fixed en	tron, Fe eported uncertain a = 2. ted errors ntal measurem	2,000 ± 6 nties represent expan

or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd ed., 1993, definition 6.10) • This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported,

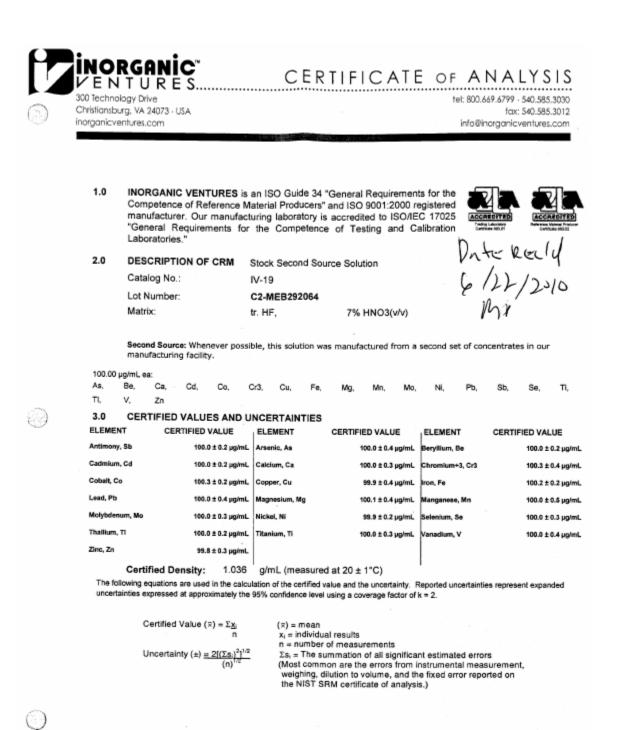
 This product is Traceable to NIST via an unbroken chain of comparisons. The uncertainties for each certified value are reported, taking into account the SRM uncertainty error and the measurement, weighing and volume dilution errors. In rare cases where no NIST SRMs are available, the term 'in-house std.' is specified.

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ki:

 $\begin{array}{ll} n & x_i = individual results \\ n = number of measurements \\ Uncertainty (\pm) = \frac{2f(\Sigma_S)^2}{(n)^{1/2}} & \Sigma_S = The summation of all significant estimated errors \\ (n)^{1/2} & (n)^{1/2} & (n)^{1/2} & \Sigma_S = The summation of all significant estimated error significant estimated error reported on the NIST SRM certificate of analysis.) \end{array}$ 



Edison



SOP No. ED-MT-016, Rev. 8 Effective Date: 03/31/2011 Page No.: 1 of 21

## Title: Mercury Analysis for Sediment and Soil Samples using the Leeman Mercury Analyzer (Cold Vapor Technique) by SW846 Method 7471A

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Approvals (Signature/Date):					
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#### 1.0 Scope and Application

#### 1.1. <u>Analytes, Matrix(s), and Reporting Limits</u>

SW846 Method 7471A is applicable to the determination of mercury in soils, sediments, bottom deposits, and sludge type materials. Mercury may be found in both inorganic and organic forms. Organomercury compounds must first be broken down to respond to the cold vapor atomic absorption technique

The typical detection limit using a 0.6 gram sample size is 0.033 mg/Kg Hg.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 2.0 Summary of Method

A digested sample is analyzed using cold vapor atomic absorption. The mercury is reduced to the elemental state and aerated from solution in a closed system. The mercury vapor passes through a cell positioned in the light path of an atomic absorption spectrophotometer. Absorbance (peak height) is measured as a function of mercury concentration.

#### 3.0 Definitions

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 4.0 Interferences

- **4.1** Potassium permanganate is added to eliminate possible interferences from sulfide.
- **4.2** Copper may also be a potential interference although no effect has been observed for samples containing up to 10 mg/l total copper.
- **4.3** Samples that contain high levels of chloride have a potential to interfere due to a reaction that takes place during the oxidation step. During this step chloride is converted to free chlorine which absorbs light at 253.7 nm. The analyst must not allow the chlorine to be swept into the optical cell. The possibility of the chlorine interfering with the analysis can be minimized by using an excess of up to 25 ml hydroxylamine hydrochloride.
- **4.4** Certain volatile organic materials that absorb at 253.7 nm wavelength may also cause interferences.

#### 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the

method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

#### 5.1. Specific Safety Concerns or Requirements

Samples that contain high concentrations of carbonates or organic material or samples that are at elevated pH can react violently when acids are added.

#### 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Mercury (1,000 PPM in Reagent)	Oxidizer Corrosive Poison	0.1 mg/M <sup>3</sup> Ceiling (Mercury Compounds)	Extremely toxic. Causes irritation to the respiratory tract. Causes irritation. Symptoms include redness and pain. May cause burns. May cause sensitization. Can be absorbed through the skin with symptoms to parallel ingestion. May affect the central nervous system. Causes irritation and burns to eyes. Symptoms include redness, pain, and blurred vision; may cause serious and permanent eye damage.
Sulfuric Acid	Corrosive Oxidizer Dehydrator Poison	1 mg/M <sup>3</sup> -TWA	Inhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.

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Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of oxnosure
Nitric Acid	Corrosive Oxidizer Poison	2 ppm-TWA 4 ppm-STEL	Signs and symptoms of exposure Nitric acid is extremely hazardous; it is corrosive, reactive, an oxidizer, and a poison. Inhalation of vapors can cause breathing difficulties and lead to pneumonia and pulmonary edema, which may be fatal. Other symptoms may include coughing, choking, and irritation of the nose, throat, and respiratory tract. Can cause redness, pain, and severe skin burns. Concentrated solutions cause deep ulcers and stain skin a yellow or yellow-brown color. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Hydrochloric Acid	Corrosive Poison	5 PPM- Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.
Potassium Permanganate	Oxidizer	5 Mg/M <sup>3</sup> for Mn Compounds	Causes irritation to the respiratory tract. Symptoms may include coughing, shortness of breath. Dry crystals and concentrated solutions are caustic causing redness, pain, severe burns, brown stains in the contact area and possible hardening of outer skin layer. Diluted solutions are only mildly irritating to the skin. Eye contact with crystals (dusts) and concentrated solutions causes severe irritation, redness, and blurred vision and can cause severe damage, possibly permanent.
1 – Always add a			eactions.
2 – Exposure lim	it refers to the	OSHA regulatory	exposure limit.

### 6.0 Equipment and Supplies

### 6.1. Instrumentation

6.1.1. Leeman Laboratories Inc. Hydra AA Automated Hg Analyzer

- **6.1.2.** Computer and Printer with Leeman WinHg software.
- 6.1.3. Analytical Balance capable of accurate weighing to 10 mg

#### 6.2. Supplies

- 6.2.1 300 ml BOD bottles
- 6.2.2 100 ml graduated cylinder
- 6.2.3 Eppendorf Pipettes and tips in various sizes
- 6.2.4 100 ml volumetric flasks
- 6.2.5 15 ml sample cups
- 6.2.6 Pump tubing:
  - Sample, viton, blue tab
  - Reductant, red tab
  - Drain, blue tab
  - Rinse, Black tab
- **6.2.7** Drying Tube Purchased pre-packed with Magnesium Perchlorate from Leeman Labs. Located prior to the optical cell.
- 6.2.8 Nitrogen supply capable of producing 80 PSI.
- **6.2.9** Autoclave capable of holding a temperature of 121°C and 15 lb (pressure) for 15 min

#### 7.0 <u>Reagents and Standards</u>

#### 7.1 <u>Reagents</u>

- **7.1.1** Sulfuric acid Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions
- **7.1.2** Nitric acid Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions
- **7.1.3** Hydrochloric acid-Concentrated (Trace Grade or Equivalent); store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.1.4** Potassium Permanganate (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.

- **7.1.5** Sodium Chloride (analytical reagent grade); store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.1.6** Hydroxylamine Hydrochloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.1.7** Stannous Chloride (ACS Grade); store at room temperature; for stability information, refer to manufacturer's instructions.
- 7.1.8 Deionized water 18 megohm minimum
- **7.1.9** 10% Hydrochloric Acid- Cautiously add 200 ml of concentrated HCl to a container and bring to final volume of 2 liters with deionized water. Store at room temperature; stable for one year.
- **7.1.10** Stannous chloride solution Add 50 g of SnCl<sub>2</sub> to 500 ml 10% HCl solution. Store at room temperature; stable for one year.
- **7.1.11** Sodium chloride/Hydroxylamine Hydrochloride solution Dissolve 120 g of NaCl and 120 g of hydroxylamine hydrochloride in deionized water and dilute to 1 liter using deionized water. Store at room temperature; stable for one year.
- 7.1.12 Potassium permanganate (KMnO<sub>4</sub>) 5% solution w/v Dissolve 100 g of KMnO<sub>4</sub> in deionized water and dilute to 2 liters using deionized water. Store at room temperature; stable for one year.

#### 7.2 Standards

Storage requirements: all standards are stored at room temperature

Shelf-life:	Stock standards – refer to manufacturer's instructions Intermediate standards – made fresh daily
	Working standards – made fresh daily
	(Note: expiration date must not go beyond the expiration date of the source stock).
Concentration:	see Attachment A for example certificates of analysis (COA) of Hg standards listed below. The COA lists the manufacturer's Lot number, certified concentration and shelf life.

Document standard preparation in the TestAmerica Edison Mercury Standard Preparation Logbook.

- **7.2.1** Stock Mercury Calibration (10 ppm Hg) Purchase from SCP Science; store at room temperature; for stability information, refer to manufacturer's instructions.
- **7.2.2** Stock Mercury Calibration Verification Standard (10 ppm Hg) Purchase from Inorganic Ventures; store at room temperature; for stability

information, refer to manufacturer's instructions.

- **7.2.3** Intermediate Calibration Standard (DCAL-Int): Dilute 1 ml of Hg calibration stock standard solution (Sec 7.2.1) to 100 ml with 0.15% HNO<sub>3</sub> The resulting solution will contain 100ppb Hg.
- **7.2.4** Intermediate Initial Calibration Verification Standard (DICV-Int): Dilute 1 ml of Hg stock Calibration Verification standard (10ppm Hg) solution to 100 ml with 0.15% HNO<sub>3</sub>. The resulting solution will contain 100ppb Hg.
- 7.2.5 Calibration Standards: Use six 300 ml BOD bottles to prepare the standards. Working in increasing order, spike the appropriate flasks with 0.0, 0.1, 1.0, 2.0, 5.0, and 10.0 ml of the Intermediate Calibration standard DCAL-INT (Sec 7.2.3). Digest the standards following Sample preparation in Sections 10.1.1 & 10.1.6. Bring the digestate to final volume of 100 mls and mix thoroughly. The corresponding final concentrations are 0.0ppb, 0.1ppb, 1.0ppb, 2.0ppb, 5.0ppb, and 10.0ppb mercury.
- **7.2.6** Initial/Continuing Calibration Verification Standard (ICV/CCV) 5.0 ppb: Spike one 300ml BOD bottle with 5ml of DICV-INT, (Sec 7.2.4). Bring the digestate to final volume of 100 mls and mix thoroughly. After digestion, the ICV/CCV will contain 5.0 ppb of Hg.

### 8.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

**8.1** Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

	Sample	Min. Sample			
Matrix	Container	Size	Preservation	Holding Time <sup>1</sup>	Reference
Soils	Glass	5 grams	Cool 4 $\pm$ 2°C	28 Days	N/A

<sup>1</sup> Inclusive of digestion and analysis.

Samples are to be analyzed without drying. A separate procedure is used to determine the percent solids in the sample.

For sample homogenization procedures refer to TestAmerica Edison SOP ED-GEN-007.

#### 9.0 Quality Control

Quality Controls	Frequency	Control Limit
Preparation Blank (PB)	1 in 20 or fewer samples	< MDL; 5% of the regulatory limit; 5% of the measured concentration in the sample
Laboratory Control Sample (LCS) in soil samples	1 in 20 or fewer samples	Vendor's certified limit
Matrix Duplicate (DUP) <sup>1</sup>	1 in 20 or fewer samples	If original sample and dup are both $\geq$ 5X RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise ±RL.
Matrix Spike (MS) <sup>1</sup>	1 in 20 or fewer samples	75-125%
Serial Dilution for 7471A	1 in 20 or fewer samples	±10%

**9.1.** <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

<sup>1</sup> The sample for DUP and MS are randomly selected, unless specifically requested by a client; Use the same environmental sample for the matrix spike and matrix duplicate sample whenever possible. If insufficient sample amount is available, another environmental sample may be used for the duplicate sample.

- **9.1.1. Preparation Blank/Method Blank:** One laboratory method/preparation blank will be analyzed with each batch of samples prepared together (not to exceed 20 samples). Preparation blank is used to identify possible contamination during acid digestion. Results must be less than the MDL, 5% of the regulatory limit for that analyte or 5% of the measured concentration in the sample. If any analyte concentration in the blank is above this control limit, the batch must be prepared again and the samples reanalyzed.
- **9.1.2.** Laboratory Control Sample Soil (LCSS): A laboratory control sample must be analyzed with each group of samples digested. For solid matrices, a vendor supplied solid matrix with certified values is carried through the same preparation procedure as the samples. The results of the solid LCS must fall within the manufacturer's certified limits for that sample. If not, all samples prepared in association with the LCS must be redigested and reanalyzed.

- **9.1.3** Matrix Duplicate (DUP): A duplicate is analyzed for each batch of samples digested. If original sample and duplicate are both ≥ RL, then 20% RPD. If original sample and duplicate are less than the RL, the RPD is not calculated; otherwise, ±RL.
- **9.1.4 Matrix Spike (MS):** A matrix spike is prepared and analyzed for each batch of samples. A portion of sample is spiked with 0.1 ug of mercury (1ml of standard DCAL-Int; Sec 7.2.3). This is equivalent to 1.0 ppb Hg (on instrument). A recovery of 75-125% is required.
- **9.1.5** Serial Dilution (SD): A five fold serial dilution must be performed on one sample per batch. The sample should contain a sufficiently high concentration; minimally a factor of 25 times the estimated detection limit. Dilute the sample by a minimum of five fold (1+4) and reanalyze. The results must agree within 10% of the original determination. If not, a chemical or physical effect should be suspected.

### 9.2. Instrument QC

- **9.2.1** Initial Calibration Verification (ICV): Initial calibration is verified after calibration using an independent check standard at a concentration near the mid-point of the calibration (5.0ppb); see Sec 7.2.6 for preparation instructions. The results must be within 10% of the true value. If it's outside of the acceptable limits, terminate the analysis, correct the problem and recalibrate the instrument.
- **9.2.2** Continuing Calibration Verification (CCV): Calibration verification is analyzed after every 10 samples and at the end of the analytical run. The CCV solution is prepared the same way as the ICV and the concentration is at the midpoint of the calibration range (5.0ppb). The results must be within 20% of the true value. If not, stop the analysis and recalibrate. Reanalyze the previous ten samples following the last good calibration verification.
- **9.2.3** Initial and Continuing Calibration Blank (ICB/CCB): ICB and CCB must be analyzed after the calibration curve, every 10 samples and at the end of the analytical run. The absolute value of the calibration verification blank must not exceed the RL. If it does, terminate the analysis, correct the problem, recalibrate and reanalyze the samples following the last good CCB. The calibration verification blank is the same blank solution as used for the calibration blank.

### 10 Procedure

- **10.1.** <u>Sample Preparation</u> (includes all samples, standards, and blanks)
  - **10.1.1** Mix the sample well and weigh approximately 0.60 grams of sample (including the LCSS) and place in the bottom of an appropriately identified 300 ml BOD bottle. For QA samples, weigh three portions of 0.60 grams of

sample and place in the bottoms of three BOD bottles labeled as SAMPLE, DUP, and MS. Spike the MS sample with 0.1 ug of mercury (1 ml of DCAL-Int standard).

- **10.1.2** Add 5 ml concentrated  $H_2SO_4$  and 2 ml concentrated  $HNO_3$  mixing well after each addition. Let stand for 10 minutes.
- **10.1.3** Add 5 ml of potassium permanganate solution to each bottle. Mix well and cover with aluminum foil. The same amount of KMnO<sub>4</sub> must be added to the standards and samples.
- **10.1.4** Set the autoclave to heat for 15 minutes at 121°C and 15 lbs pressure. Fill the reservoir with tap water. Set the autoclave to slow exhaust to prevent the BOD bottles from rupturing.
- **10.1.5** Place samples into autoclave and start cycle. Be sure to take the samples out of the autoclave when the pressure gauge reads zero. This will eliminate the possibility of back pressure that will fill the reservoir with steam exhaust water. Note: Be CAREFUL the autoclave gets VERY HOT. Cool samples down before taking them out.
- 10.1.6 Cool and dilute to volume of 100 ml with deionized water. Add 6 ml Sodium chloride - Hydroxylamine hydrochloride solution to reduce excess permanganate. Mix well; solution should become colorless. If necessary additional Sodium chloride - Hydroxylamine HCl solution may be added. Note: if additional Sodium chloride – Hydroxylamine HCl is necessary, then add the same volume of that reagent to all the BOD bottles associated with that batch. Wait at least 30 seconds after decolorization before analyzing.

### 10.2. Calibration

- 10.2.1 The instrument must be calibrated daily or once every 24 hours and each time the instrument is set up. The instrument is calibrated according to the manufacturer's specifications and must contain at least four standards and a blank. The laboratory currently uses five standards and a blank. The correlation coefficient of the calibration curve must be ≥0.995. If it does not, the problem must be corrected, and the instrument must be recalibrated. Standard preparations must be documented in the Standard Preparation Logbook located in the Mercury analysis room.
- **10.2.2** Prepare the calibration standards and Calibration Verification Standards as stated in Sections 7.2.5 & 7.2.6.

### 10.3. Sample Analysis

**10.3.1** Following a sample digestion procedure, the samples are ready for instrumental analysis. It is advisable to investigate each matrix for any

complexities, which might adversely affect the acquisition of valid data.

**10.3.2** The following analytical run sequence is currently used for samples analyzed under Method 7471A:

Instrument Calibration (Blank and five standards) ICV ICB 10 Samples CCV CCB 10 Samples CCV CCB Repeat until run is complete CCV CCB

**10.3.3** Instrument Operation:

**10.3.3.1** Turn on Computer, Printer and Monitor.

- **10.3.4** Plumbing the Reagent Lines:
  - **10.3.4.1** One at a time, feed each of the pump tubes into a pump cassette, sliding the tube through the plastic clips at the bottom until the plastic tab is secure. Then, holding the tube taut, slide the loaded cassette onto the pump head and click the clamp, lever up. The tab end of the tube should be located at the front of the pump head.
  - **10.3.4.2** Reductant (Red); Connect tab end of tube to the reductant bottle and the other end to the bottom of the mixing tee.
  - **10.3.4.3** Sample (Blue); Connect tab end of tube to the autosampler probe and the other end to the top of the mixing tee.
  - **10.3.4.4** Drain (Blue) Connect the tab end of tube to the sample discharge tube connected on the Liquid/Gas separator and the other end to the waste line.
  - **10.3.4.5** Rinse (Black): Connect tab end of tube to rinse tubing that is connected to the rinse bottle. Connect the other end to the rinse tubing leading to the rinse cup.
- **10.3.5** Preparation of Reagents:
  - **10.3.5.1** Pour the SnCl<sub>2</sub> solution into the reductant bottle and connect to the red reductant tube connector.
  - **10.3.5.2** Pour the ten percent HCl solution into the Rinse reservoir

bottle.

- **10.3.6** Starting Program:
  - **10.3.6.1** Double click WinHg icon on the desktop. This will open the WinHg Runner 1.5 window.
- **10.3.7** Selecting Protocol/Method:
  - **10.3.7.1** Go to the Protocol box in the upper left hand corner and click on the "▼" button.
  - **10.3.7.2** Scroll down to the desired protocol and click on it, (i.e. SW846A).
- **10.3.8** Creating a New Data Set/File Name:
  - **10.3.8.1** From the drop down file menu, click on 'New Data Set'.
  - **10.3.8.2** Type in name of Data Set (i.e. 15990HG1), and click Ok, this opens new batch name box.
  - **10.3.8.3** Type in Batch name (i.e. 15990HG1), and click OK.
  - **10.3.8.4** The new Data Set and batch name should now appear in the respective locations.
- **10.3.9** Opening Data Base Window:
  - 10.3.9.1 From the tool bar click on the "DB↓" button. Check to ensure that the correct protocol is open in the Protocol Box (in upper left corner) of the Data Base window. The Dataset/Protocol in the upper right hand corner, should match that in the Runner Window, (i.e. 5990HG1/SW846A).
  - **10.3.9.2** Report specs should also have the same protocol open.
  - **10.3.9.3** Left click the Apply button.
  - **10.3.9.4** Left click the Cal Curve tab to view calibration values.
- **10.3.10** Turning on Instrument (Lamp/Gas/Pump):
  - **10.3.10.1** Return to the Win Hg Runner window by clicking anywhere inside of runner window.
  - **10.3.10.2** In main 'WinHg Runner' window, click on Control tab.
  - **10.3.10.3** Under Lamp click "ON" to turn lamp on. Lamp should warm up for approximately 5 minutes before analyzing any samples or

standards.

- **10.3.10.4** Under Pump click "ON" to turn pump on. Click on "Standby" to keep lamp on while instrument is not running, otherwise lamp will automatically shut off after 15-20 minutes.
- **10.3.11** Setting up rack:
  - **10.3.11.1** In 'WinHg Runner' window, click on icon that looks like rack. This will open up the rack editor.
  - 10.3.11.2 Type in the Sample IDs under the 'Extended ID' column starting with sequence 1 and follow down until sequence 44. The dilution factor for the sample is typed in under the "Extended ID" column. "Weight" and "Volume" column should always have a value of 1.00 for all samples; the actual weight and volume will be included later in the calculation and data processing. The last column is the macro column. This is where the analyst tells the instrument to automatically go to the check standard cups (i.e. C3 C1 CP). Click on "File", "Save As;" use the prep batch number as the save file name.

Cup SampleID	ExtendedID	Weight	Volume	Macros
1	PBS011509	1.00	1.00	C3 C1 CP
2	LCSS	1.00	1.00	
3	610544	1.00	1.00	
4	610544D	1.00	1.00	
5	610544MS	1.00	1.00	
10	 610545@10	1.00	1.00	C2 C1
10	610545@10	1.00	1.00	02.01

The Macro command C3 C1 CP tells the instrument to run the check standards before running the sample cup. The Macro command C2 C1 tells the instrument to run the sample cup first then the check standards. Make sure the CCV/CCB is run so no more than 10 samples run between check standards.

- **10.3.12** Activating and Running Standards:
  - **10.3.12.1** Click on the Standards tab in the Runner Window.
  - **10.3.12.2** Click on button to activate standard cup S1 (0.0ppb), S2 (0.1ppb), S3 (1.0ppb), S4 (2.0ppb), S5 (5.0ppb), S6 (10.0ppb) and Rep1. Fill cups appropriately.
  - **10.3.12.3** Click on C1<CCB>, C2<ICV/CCV>, C3<ACCV>, C4<ACCV> Click New Cal Reset "OK"
  - **10.3.12.4** Turn gas and pump on. Click on Standard Auto to run

standards.

- **10.3.12.5** Check the calibration curve in the 'Database' window. If the correlation coefficient (Rho) of the calibration is ≥0.995, it is acceptable; Click Accept. Recalibrate if Rho is <0.995.
- **10.3.12.6** Print the calibration curve by hitting the printer icon.
- **10.3.12.7** In the 'WinHg Runner 1.5' window, open File from the drop down menu, Choose Page eject, to print the standards on their own separate page. Click Report and Clear.
- **10.3.13** Analyzing Samples:
  - **10.3.13.1** Click on the Sample tab in the 'WinHg Runner 1.5' window.
  - 10.3.13.2 Select the rack you want to run by clicking the "▼" button for Rack Name and highlighting the desired rack, for station 1 and station 2. If you're only running one rack, make sure you highlight the blank space at the top of the rack list for the unused station.
    - **10.3.13.2.1** Enter the start cup and the end cup values. If you're only running one rack, make sure the start and end cup are blank for the empty station.
    - **10.3.13.2.2** Real time print should be checked.
    - **10.3.13.2.3** Pour samples into polypropylene test tubes and place them in the appropriate sequence noted on rack. Rack sequence is from right to left i.e. (.... 3, 2, 1).
    - **10.3.13.2.4** Click Run Auto to begin analyzing samples.
- **10.3.14** Printing Report and Post-Run Report:
  - **10.3.14.1** In the 'WinHg Runner 1.5' window, click File, Page eject to print out last page of data.
  - 10.3.14.2 In the 'WinHg Data Base' window, click on the "Report tab."
  - **10.3.14.3** Mark the box next to the Batch ID with a check (✓) by clicking on the box. The sample IDs should now show up under the Record ID column.
  - 10.3.14.4 Select the IDs you wish to print by checking on the box in the far left column of the desired sample, a check mark (✓) on the box will include data in report, a box without a checkmark will exclude the sample data from the report. You can select all of them by checking the ALL button, or only a few by checking the

NONE button and then clicking the box next to the desired sample.

- 10.3.14.5 Under the Report Specs, click the "▼" button to the right of the box and select the report format you wish to use.
  e.g. SW846 = results in concentration, and SW846int = results in intensities.
- **10.3.14.6** Click the include button under the Select Records heading.
- **10.3.14.7** Click the Generate Report button at the lower right hand corner. This will activate the 'Generate Report' window.
- **10.3.14.8** To send report to the printer, choose the report option, under the format heading along with the printer option under destination. Click 'Generate.'
- **10.3.15** Creating a PRN File:
  - **10.3.15.1** Choose the PRN option under format.
  - **10.3.15.2** Choose the Disk File option under destination (should default to this option).
  - 10.3.15.3 The output file path and name is: F:\INORG\LEEMAN#\DATASET NAME. You must delete C:\ before typing in path. Click 'Generate.'
- **10.3.16** Shutting Down the Instrument:
  - **10.3.16.1** Close the 'WinHg Database' window.
  - **10.3.16.2** Open the control tab in the 'WinHg Runner 1.5' window.
  - **10.3.16.3** Click the "Off" button for the lamp, pump, and gas.
  - **10.3.16.4** Close the WinHg Runner 1.5 window.

#### 11.0. Calculations / Data Reduction

**11.1.** Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

**11.2.** Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

**11.3.** Concentration: (mg/Kg) =  $\frac{C \times V1 \times D}{W}$ 

Where: C= Element concentration from instrument (ppb) V1= Final volume of sample digested (in liters) D= Dilution performed on sample W= Initial weight of sample digested (in gram)

**NOTE:** All dry weight corrections are made in LIMS at the time the final report is prepared.

- **11.4.** Data Processing:
  - **11.4.1.** All data is recorded directly in TALS' Analyst Desktop II program.
  - 11.4.2. Import Data from WinHG Leeman to TALS
    - 11.4.2.1. Start WinHg and Select 'DataBase'
    - **11.4.2.2.** Select Report Tab and Batch ID
    - **11.4.2.3.** Select 'Generate Report,' and PRN File format
    - **11.4.2.4.** Select Output File Path under c:\Hg\RawData\TodaysDate.PRN
    - 11.4.2.5. Select 'Generate'
  - **11.4.3.** Sample and standard preparations must be documented in the Analyst Desktop II program located in TestAmerica Laboratory System (TALS). The analyst must enter the following information: Source standard, Initial and final sample volume, spike name and amount used and all reagents and their corresponding lot numbers, creation and expiration dates.
  - **11.4.4.** All reagents must be recorded in the Hg Reagent Prep Log.
  - **11.4.5.** Metals Data Review checklist (ED-WI-007) must be filled out prior to data submission.

#### 12.0. Method Performance

#### 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

#### 12.2 Instrument Detection Limit

The IDL for each analyte must be determined for each wavelength used on each

instrument. The IDL must be determined annually or if the instrument is adjusted in any way that may affect the IDL. The IDL is determined by multiplying the average of the standard deviations obtained from the analysis of seven reagent blanks by 3.14.

#### 12.3 Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 12.4 Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

#### 13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention.

#### 14.0. Waste Management

- **14.1.** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).
- **14.2.** The following waste streams are produced when this method is carried out:

•Digested Samples: Corrosive Acid- Materials that are not above regulatory limits will be submitted for elementary neutralization with 50% sodium hydroxide solution (Siedler Chemical SC-1824-03). Major concern is heat generated from the neutralization process. Initial volume of acid waste to be neutralized should be no more than 15 gallons. Finished neutralization with sodium bicarbonate (Siedler Chemical SC-0219-25) to a pH of 6 – 9 in the primary tank. Once pH has been established the primary tank is transferred through filter housing to a secondary tank. The pH is rechecked. If the pH is within specifications, the secondary tank is released to the municipal sewer system.

•Samples above regulatory limits and expired RCRA metals standards (Waste Corrosive Liquid, Acidic, Inorganic, n.o.s.) are collected in satellite accumulation and sent off site through a Waste disposal vendor.

Onyx Profile WIP Number: 590598 Teris Profile Number 50016653

#### 15.0. <u>References / Cross-References</u>

- **15.1.** <u>Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846)</u>, Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996.
- **15.2.** Leeman Leeman Hydra AA Operating Manual.
- **15.3.** TestAmerica Edison Document ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.4.** Corporate Environmental Health and Safety Manual CW-E-M-001, most current revision.
- **15.5.** TestAmerica Edison Subsampling SOP, ED-GEN-007, most current revision.
- **15.6.** TestAmerica Edison SOP ED-GEN-022, *Training*, most current revision.
- **15.7.** TestAmerica Edison SOP No. ED-SPM-008, *Laboratory Waste Disposal Procedures*, current revision
- **15.8.** Metals Data Review Checklist Work Instruction # EDS-WI-007, most current revision.

#### 16.0. <u>Method Modifications:</u>

ltem	Method #	Modification
10.1.6	7471A	Stannous Chloride is automatically added via the instrument versus the manual addition of Stannous Chloride as stated in the method. This is an instrument manufacturer's improvement that will reduce error due to loss of Mercury.

#### 17.0. Attachments

Attachment 1: Certificate of Analysis of stock standards

#### 18.0. <u>Revision History</u>

- Revision 8, dated 31 March 2011
  - Sec 3: Updated LQM reference for the list of definitions.
  - Sec 7.2.6: Clarified preparation procedure for ICV and CCV; both QC samples are prepared from the same second source stock standard; Deleted Sec 7.2.7 (CCV standard preparation procedure).
  - Sec 9.2.2: Revised to reflect actual lab procedures.
  - Sec 9.1.3: Expanded the criteria for Matrix Duplicate.
  - Sec 9.2.3: Revised the pass/fail criteria for ICB/CCB to reflect actual lab practices.
  - Sec 11.4: Updated data reduction procedure in accordance with new TALS.

- Sec 15: Added applicable references.
- Revision 7, dated 17 March 2009
  - Sec. 6.2: Added autoclave to list of supplies.
  - Deleted Potassium Persulfate in reagent list (Sec 7.1) and in the primary material used (Sec 5.2); not required for method 7471A.
  - Sec 7.2 Standards: Revised the Hg stock standard concentrations and preparation of standards.
  - Deleted LCSS-D (LCS Duplicate) and Blank Spike as QC samples.
  - Sec 9.1.5: Added Serial Dilution in QC type.
  - Sec 10.1.6: Added text: if additional Sodium chloride Hydroxylamine HCl is necessary, then add the same volume of that reagent to all the BOD bottles associated with that batch.
  - Sec 6.2.6 & Sec 10.3.4.5: Added 'Rinse Black' and changed Drain 'Black' to Drain 'Blue' in the list of Reagent lines.
- Revision 6, dated 08 October 2007
  - Section 12.1 Calculation. The 1000 conversion factor is not required in the calculation of final results, therefore deleted.

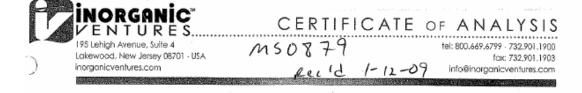
### Attachment 1

Certificate of Analysis						
Catalogue Number : Description :	141-110-111/141-110-112/141-110-115 PlasmaCAL - ICP-MS Verification Standard 1 Solution B					
Lot Number : Expiration Date :	Solution B SC8298812 January 2010					
Analysis of Solution Standard by Inductively Coupled Plasma Spectroscopy (ICP-AES) traceable to NIST Standard Reference Materials : 3133						
Act	ual Concentrations					
Hg : 9.98 μg/ml						
Matrix Densi						
MS 0882						
MS 0882 Rec d 1/14/09						
Certified by :	Certification Date : October 28, 2008					
Thomas Znoj, Chemistry Mar						
This ICP-AES & ICP-MS Standard is guaranteed to be stable and accurate to within plus or minus 1.0% of the actual concentration up to the expiry date, provided the solution is kept tightly capped and stored under normal laboratory conditions. For these solutions, 18 megohm/cm double deionized water, high-purity acids, Class A glassware and acid- cleaned bottles are used. The Material Safety Data Sheet and this Certificate of Analysis are available on our web site. (Également disponible en Français)						
	9001:2000 Quality System and ISO 17025 (in-process) SCP SCIENCE am, Bale D'Urfé, QC, Canada H9X 486					

Phone : (514) 457-0701 Fax : (514) 457-4499 Web Site: www.scbscience.com



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1.0 INORGANIC VENTURES is an ISO Guide 34 "General Requirements for the Competence of Reference Material Producers" and ISO 9001:2000 registered manufacturer. Our manufacturing laboratory is accredited to ISO/IEC 17025 "General Requirements for the Competence of Testing and Calibration Laboratories."



 2.0
 DESCRIPTION OF CRM
 10 μg/mL Mercury in 10% (v/v) HCL

 Catalog Number:
 MSHG-10PPM

 Lot Number:
 B2-HG02061

 Starting Material:
 Hg metal

 Starting Material Purity (%):
 99.999549

 Starting Material Lot No:
 05214TX

 Matrix:
 10% (v/v) HCL

#### 3.0 CERTIFIED VALUES AND UNCERTAINTIES

Certified Concentration: 10.027 ± 0.020 µg/mL

Certified Density:

)

#### 1.019 g/mL (measured at 22° C)

The following equations are used in the calculation of the certified value and the uncertainty. Reported uncertainties represent expanded uncertainties expressed at approximately the 95% confidence level using a coverage factor of k = 2.

Certified Value  $(x) = \sum \underline{x}_i$ n Uncertainty  $(\pm) = \underline{2[(\sum \underline{s}_i)^2]^{1/2}}{(n)^{1/2}}$ 

#### 4.0 TRACEABILITY TO NIST AND VALUES OBTAINED BY INDEPENDENT METHODS

"Property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually
national or international standards, through an unbroken chain of comparisons all having stated uncertainties." (ISO VIM, 2nd
ed., 1993, definition 6.10)

This product is Traceable to NIST via an unbroken chain of comparisons to the following NIST SRMs:



SOP No. ED-GCS-003, Rev. 6 Effective Date: 06/09/2008 Page No.: 1 of 28

## Title: ANALYSIS OF ORGANOCHLORINE PESTICIDES BY GAS CHROMATOGRAPHY, [SW846 Method SW8081A]



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#### Edison

Distributed To:

#### 1.0 Scope and Application

#### 1.1 <u>Analytes, Matrix(s), and Reporting Limits</u>

Method 8081 is used to determine the concentrations of various organochlorine pesticides in extracts from solid and liquid matrices, using dual fused-silica, opentubular, capillary columns with electron capture detectors (ECD). The list of analytes and their corresponding reporting limits are as follows:

Parameter		Soil	Water	Leachate
	CAS Registry No.	Reporting Limits (ug/L)	Reporting Limits (ug/L)	Reporting Limits (ug/L)
Aldrin	309-00-2	6.7	0.050	
Alpha-BHC	319-84-6	6.7	0.050	
Beta-BHC	319-85-7	6.7	0.050	
Delta-BHC	319-86-8	6.7	0.050	
Gamma-BHC (Lindane)	58-89-9	6.7	0.050	0.00050
Chlordane	57-74-9	67	0.50	0.0050
4,4' –DDD	72-54-8	6.7	0.050	
4,4' –DDE	72-55-9	6.7	0.050	
4.4' –DDT	50-29-3	6.7	0.050	
Dieldrin	60-57-1	6.7	0.050	
Endosulfan I	959-98-8	6.7	0.050	
Endosulfan II	33213-65-9	6.7	0.050	
Endosulfan sulfate	1031-07-8	6.7	0.050	
Endrin	72-20-8	6.7	0.050	0.00050
Endrin aldehyde	7421-93-4	6.7	0.050	
Endrin ketone	53494-70-5	6.7	0.050	
Heptachlor	76-44-8	6.7	0.050	0.00050
Heptachlor epoxide	1024-57-3	6.7	0.050	0.00050
Methoxychlor	72-42-5	6.7	0.050	0.00050
Toxaphene	8001-35-2	67	.50	0.0050
Gamma- Chlordane	5103-74-2	6.7	0.050	
Alpha-Chlordane	5103-71-9	6.7	0.050	

The most current MDLs and RLs for this method can be found in TestAmerica Edison Work Instruction No. EDS-WI-072, *SW846 Method 8081A, Current MDLs and Reporting Limits*, (most current revision).

**1.2** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and Section 20 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 2.0 <u>Summary of Method</u>

- **2.1.** Samples undergo a preparation step prior to analysis by SW846 Method 8081A. A measured volume or weight of sample (15g for soil, 1 g for waste, 1000 ml for water, and 100 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique. (Reference the applicable Organic Sample Prep SOPs listed below). The effective final volume is usually between 5 and 20 ml in hexane.
  - **2.1.1.** Aqueous samples are extracted using SW846 Method 3510C (SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel*).
  - **2.1.2.** Solid samples are extracted using SW846 Method 3550B: Sonication (SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction) or SW846 Method 3541 (SOP No. ED-ORP-016: Automated Soxhlet Extraction of Solid Samples Pesticides/PCBs).
  - **2.1.3.** Organic liquids are prepared using SW846 Method 3580A (SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs).
  - 2.1.4. Extract cleanup steps are employed depending on the nature of the matrix interferences. Suggested cleanups include SW846 Method 3620B (SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts) and SW846 Method 3660B (SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts).
- **2.2.** After cleanup, the extract is analyzed by injecting a 2-uL sample into an Agilent Technologies gas chromatograph equipped with a dual wide-bore fused silica capillary columns and electron capture detectors (GC/ECD).
- **2.3.** For pesticide analysis a system performance check (DDT/Endrin breakdown) and a calibration verification standard must be run prior to analysis. Failure of either will generally indicate the need for injection port/column maintenance and/or recalibration.
- **2.4.** Samples are analyzed after all the necessary checks have been performed. Samples analyzed for pesticides require an additional post analysis Quant Report to be printed and attached to the chromatographic report.
- **2.5.** All samples are then manually reviewed. Secondary column confirmation of target compounds and quantification are conducted by the analyst as required.

#### 3.0 Definitions

For a complete list of definitions refer to Appendix 5 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 4.0 Interferences

- **4.1.** Interferences by phthalate esters introduced during sample preparation can pose a major problem in pesticide determinations.
  - **4.1.1.** Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- **4.2.** The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting organochlorine pesticides. Sulfur contamination should be expected with sediment samples. If sulfur is encountered, employ the sulfur removal procedures detailed in SW846 Method 3660B (SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts). Note that the recover of Endrin aldehyde is adversely affected by the TBA cleanup procedure detailed in this method. Accordingly, this compound must be determined prior to sulfur cleanup.
- **4.3.** Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts)).
  - **4.3.1.** Check Florisil prior to use to assure quantitative recovery of targeted analytes. Duplicate checks are required for each new lot or every three hundred samples whichever is more frequent.
  - **4.3.2.** Check Florisil by spiking 1ml of the Pest Mix midpoint (Supelco Catalog No. 47426) and 0.5 ml of trichlorophenol (Protocol Catalog No. S-3640) onto the cartridge and concentrating to final volume of 1 ml. Inject 1 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.

#### 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

### 5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

#### 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure			
Acetone	Flammable	1000 ppm-TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.			
Hexane	Flammable Irritant	500 ppm- TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.			
Methanol	Flammable Poison Irritant	200 ppm- TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.			
Methylene ChlorideCarcinogen Irritant25 ppm- TWA 125 ppm- STELCauses irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.						
	1 – Always add acid to water to prevent violent reactions.					
2 – Exposur	2 – Exposure limit refers to the OSHA regulatory exposure limit.					

### 6.0 Equipment and Supplies

#### 6.1. Instrumentation:

- **6.1.1.** Gas Chromatograph: The system used is an HP and an Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC). Each GC is equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual megabore capillary columns that are suitable for the analysis of organochlorine pesticides. All operations are as automated as possible with the equipment utilized.
- **6.1.2.** Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles samples between the sample tray and the injector turret.
  - **6.1.2.1.** The samples are injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the ramp pressure mode.
  - **6.1.2.2.** Liners: The injection port is each fitted with replaceable, heavy-walled siltek-coated glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.
  - **6.1.2.3.** Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35°C and 350°C.
    - **6.1.2.3.1.** Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column is used for sample quantitation. The secondary confirmation column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column.
  - **6.1.2.4.** Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.
    - **6.1.2.4.1.** Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5) is fed from a supply other than the injection port.

### 7. Reagents and Standards

#### 7.1. Reagents

- **7.1.1.** Gases: Ultra high purity (99.999%) Helium is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity (99.999%) Argon (95%) / Methane (5%) (a.k.a. P-5 Mixture) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. They are supplied by M-G Industries (Valley Forge, PA). Both gases are supplied at tank pressures of 2000-2400 psig., for a 300 cft. tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.
  - **7.1.1.1.** The gas streams are polished using three traps or filters before introduction to the GC The traps are as follows:
    - 7.1.1.1.1. Hydrocarbon trap
    - **7.1.1.1.2.** H<sub>2</sub>O trap
    - **7.1.1.1.3.** O<sub>2</sub> scrubber
  - **7.1.1.2.** Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).
- **7.1.2.** Solvents used in the extraction and cleanup procedures include n-hexane, methylene chloride, and acetone that are exchanged to n-hexane prior to analysis.
- **7.1.3.** Hexane is required in this procedure. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

#### 7.2. Standards

**7.2.1.** Standards are purchased as concentrated solutions (see Section 7.2.2).

NOTE: Independent sources are used for quantitation standards and spiking standards

**7.2.1.1.** Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent as described in Section 7.2.2.1.

7.2.2.	Standard	mixes	and	sources	*.
					-

Standard Name	Source			
CLP Organochlorine Pesticide	Supelco Catalog No. 47426-U			
Mix				
SS CLP Organochlorine Pesticide	Supelco Catalog No 4S726-U (second			
Mix	source)			
Tetrachloro-m-xylene (TCmX)	Supelco Catalog No 48460			
and Decachlorobiphenyl (DCB)				
Surrogates Mix				
Tetrachloro-m-xylene (TCmX)	Supelco Catalog No 861275			
and Decachlorobiphenyl (DCB)				
Surrogates Spike Mix				
Endrin/DDT	Supelco Catalog No 48282			
Chlordane	Supelco Catalog No 48065U			
Toxaphene	Supelco Catalog No 48103			
TCLP Pesticide Spiking Mix 1	Accustandard Catalog No. TCLP-PES-1			
TCLP Pesticide Spiking Mix 2	Accustandard Catalog No. TCLP-PES-2			
*Suppliers with equivalent standards may be substituted				

\*Suppliers with equivalent standards may be substituted..

The components of each standard mix are as follows:

Parameter	Supelco Standard Catalog No.	Concentration of Standard (ug/ml)
Aldrin	47426 & 4S726	2000
Alpha-BHC	47426 & 4S726	2000
Beta-BHC	47426 & 4S726	2000
Delta-BHC	47426 & 4S726	2000
Gamma-BHC (Lindane)	47426 & 4S726	2000
Alpha -Chlordane	47426 & 4S726	2000
Gamma -Chlordane	47426 & 4S726	2000
4,4' –DDD	47426 & 4S726	2000
4,4' –DDE	47426 & 4S726	2000
4.4' –DDT	47426 & 4S726	2000
Dieldrin	47426 & 4S726	2000
Endosulfan I	47426 & 4S726	2000
Endosulfan II	47426 & 4S726	2000
Endosulfan sulfate	47426 & 4S726	2000
Endrin	47426 & 4S726	2000
Endrin aldehyde	47426 & 4S726	2000
Endrin ketone	47426 & 4S726	2000
Heptachlor	47426 & 4S726	2000
Heptachlor epoxide	47426 & 4S726	2000
Methoxychlor	47426 & 4S726	2000
Toxaphene	48103	2000
Tetrachloro-m-xylene (TCmX)	48460	200
Decachlorobiphenyl (DCB)	48460	200

Parameter	Supelco Standard Catalog No.	Concentration of Standard (ug/ml)
Tetrachloro-m-xylene (TCmX)	861275	10
Decachlorobiphenyl (DCB)	861275	10

#### 7.2.2.1. Standards Preparation

7.2.2.1.1. Calibration Mix (CLP Organochlorine Pesticide Mix)

The 5 point calibration standards are prepared as detailed in the following table using volumetric glassware and hexane as the diluent:

Initial Calibration Standards Prep (CLP Organochlorine Pesticide Mix)					
Stock Std	10 ppb	50 ppb	100 ppb	250 ppb	500 ppb
CLP Organochlorine	Volume	Volume	Volume	Volume	Volume
Pesticide Mix (2000	brought up to	brought up	brought up to	brought up to	brought up to
ug/ml)	200 ml	to 200 ml	500 ml	200 ml	200 ml
Supelco 47426-U	hexane:	hexane:	hexane:	hexane:	hexane:
	1.0 ul	5.0 ul	25 ul	25 ul	50 ul
Initial (	Calibration Star	ndards Prep (	TCMX/DCB Sui	rogate Mix)	
Stock Std	25 ppb	50 ppb	100 ppb	150 ppb	200 ppb
Tetrachloro-m-xylene/	Volume	Volume	Volume	Volume	Volume
Decachlorobiphenyl	brought up to	brought up	brought up to	brought up to	brought up to
Surrogates Mix	200 ml	to 200 ml	500 ml	200 ml	200 ml
(200 ug/ml)	hexane:	hexane:	hexane:	hexane:	hexane:
Supelco 48460	25 ul	50 ul	250 ul	150 ul	200 ul

- **7.2.2.2.** Pesticide Surrogate Spike Mix (10 ug/ml) : DCB & TCMX Mix Solution, custom blend in final volume of 50 ml (Acetone), Supelco Catalog No. 861275 . Used as received from Supelco.
- 7.2.2.3. Surrogate Oil Spiking Solution (0.5 ug/ml DCB &TCMX) : Prepared by bringing 2.5 ml of Tetrachloro-m-xylene (TCmX) and Decachlorobiphenyl (DCB) Surrogates Spike Mix (Supelco Catlog No. 861275) up to final volume of 50 ml Acetone. This solution is used for surrogate spiking of samples prepped via TestAmerica Edison SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A, most current revision.
- **7.2.2.4.** Pesticide Spiking Solution (20 ug/ml): Bring 1.0 ml of CLP Organochlorine Pesticide Mix (Supelco Catalog No. 47426-U -2000 ug/ml) up to a final volume of 100 ml with Acetone.

- 7.2.2.5. Pesticide Oil Spiking Solution (2 ug/ml) Bring 1.0 ml of Pesticide Spiking Solution (20 ug/ml, see Section 7.2.2.4) to a final volume of 10 ml Acetone. This solution is used for surrogate spiking of samples prepped via TestAmerica Edison SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A, most current revision.
- 7.2.2.6. TCLP Pesticide Spiking Solution (5 ug/ml single response Pesticides, 25 ug/ml Chlordane and 50 ug/ml Toxaphene): Add 0.025 ml of TCLP Pesticide Spiking Mix 1 (2000 ug/ml, Accustandard Catalog No. TCLP-PES-1) and 0.125 ml of TCLP Pesticide Spiking Mix 2 (2000 ug/ml Chlordane and 4000 ug/ml Toxaphene, Accustandard Catalog No. TCLP-PES-2) to a volumetric flask and dilute to a final volume of 10 ml Acetone.
- **7.2.2.7.** System Performance Solution (Breakdown Check) (0.250 ug/ml): The breakdown check is prepared by taking 250 ul of 500 ug/ml DDT/Endrin Mix (Supelco Catalog No 48282) and bringing it up to a volume of 500 ml with hexane
- 7.2.2.8. Chlordane Calibration Solution (1000 ppb solution w/ surrogates TCMX & DCB at 100 ppb): 100 ul of 1000 ug/ml chlordane stock (Supelco Catalog No 48065U) and 50 ul of 200 ug/ml surrogate stock (Supelco Catalog No 48460). Dilute to 100 ml in Hexane. <u>NOTE</u>: Midpoint calibration standard only for multiple responders.
- 7.2.2.9. Toxaphene Calibration Solution (1000 ppb solution w/surrogates TCMX & DCB at 100 ppb) : 100 ul of 1000 ug/ml Toxaphene stock (Supelco Catalog No 48103) and 50 ul of 200 ug/ml surrogate stock (Supelco Catalog No 48460). Dilute to 100 ml in Hexane. <u>NOTE</u>: Midpoint calibration standard only for multiple responders

# 8. Sample Collection, Preservation, Shipment and Storage

**8.1.** Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml	Cool 4 <u>+</u> 2°C	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 <u>+</u> 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

# 9. Quality Control

9.1. Sample QC - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) <sup>1</sup>	1 in 20 or fewer samples	Statistical Limits 4
Matrix Spike (MS) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits 4
MS Duplicate (MSD) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits 4
Surrogates	every sample <sup>3</sup>	Statistical Limits <sup>4</sup>

<sup>1</sup> LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract. <sup>2</sup> The sample selection for MS/MSD are randomly selected, unless specifically requested by a

client....predetermined by the extraction lab.

<sup>3</sup> Analytical and QC samples (MB, LCS, MS/MSD)

<sup>4</sup> Statistical control limits are updated annually and are updated into LIMS.

- **9.1.1.** Method Blanks are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.
- 9.1.2. Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the

LCS must fall within lab generated acceptance criteria.. If the spiked sample recovery results fall outside the laboratory generated limits (refer to TestAmerica Edison Work Instruction No. EDS-WI-017), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QA batch must be re-extracted and reanalyzed.

- **9.1.3. Matrix Spike/Matrix Spike Duplicate (MS/MSD):** A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to TestAmerica Edison Work Instruction EDS-WI-017, EPA Method 8081A Current Spike, RPD, Surrogate Limits). If the MS/MSD recovery limits fall outside of lab limits the LCS recovery is evaluated and corrective action is taken as described in 9.1.2.
- **9.1.4. Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (refer to TestAmerica Edison Work Instruction EDS-WI-017, *EPA Method 8081A Current Spike, RPD, Surrogate Limits*). If both TCMX and DCB recovery are outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as "estimated concentration".

# 9.2. Instrument QC

### 9.2.1. GC System Performance Check

- **9.2.1.1.** Endrin/4,4'-DDT Breakdown: Prior to performing any standards or sample analysis, a daily check is made on the chromatographic performance of the system. This performance check is made by injecting a standard of Endrin and DDT, each at a 250-ppb level (see Section 7.2), and calculating the percentage breakdown for each compound.
- 9.2.1.2. Ideally, only two peaks will be seen (one for Endrin and one for DDT). As a rule, this is not the case. It is normal to observe up to six peaks. Three peaks are attributable to Endrin and its degradation products: Endrin Aldehyde (EA) and Endrin Ketone (EK). Three peaks are attributable to DDT and its degradation products: DDE and DDD. Calculate the percentage breakdown as follows:

#### Endrin:

(Areas of EA + EK) (Areas of EA + EK + Endrin) X 100 = % breakdown Endrin

DDT:

(Areas of DDE + DDD) (Areas of DDE + DDD + DDT) X 100 = % breakdown DDT

- **9.2.1.3.** If the percentage breakdown for either Endrin or DDT is greater than 15%, the system <u>CANNOT</u> be used for pesticide analysis. If the Endrin/DDT performance check fails injection port/column maintenance must be performed. Usually, changing the glass wool/liner will cure most breakdown problems in the injection port. Depending upon the nature of the samples, the entire injection port will occasionally need to be cleaned. This cleaning is best done with 1:1 Acetone: Hexane. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column. (Note: the septa should be changed each time the injection port is opened).
- **9.2.1.4.** After injection port/column maintenance has been performed, and the columns have been given time to equilibrate (baseline back down to normal) the Endrin/DDT must be re-injected and the system performance re-evaluated.

# 9.2.2. Initial Calibration Range and Initial Calibration Verification (ICV)

- **9.2.2.1.** *Initial Calibration Range:* Single component pesticides are calibrated using a five-point calibration range. Multi-component pesticides are calibrated using a single point calibration at the anticipated midpoint of the calibration range. Standards are prepared following the instructions in Section 7.2.
- **9.2.2.2.** Single response Pesticide Calibration: All single component pesticides and two surrogates are calibrated with a minimum of 5 concentrations. Single component pesticides are analyzed at 10, 50, 100, 250 and 500 ppb. Surrogate standards are analyzed at 25, 50, 100, 150 and 200 ppb. See Section 7.2 for details on standard prep.
- **9.2.2.3.** Multi-response Pesticide Calibration: Chlordane (technical) and Toxaphene initial calibration is accomplished by analysis of a single point at 1000 ppb (see Section 7.2 for details on standard prep).
- **9.2.2.4.** *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2 and must be from a source separate from the standards used in the Initial Calibration Range.

**9.2.3.** Continuing Calibration Verification (CCV): For single component pesticides, a mid-point Continuing Calibration Verification (CCV) must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

### 9.2.4. Calibration Acceptance Summary

- **9.2.4.1. Retention Time Windows:** Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. All gas chromatographs used for pesticides analysis at TestAmerica Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.
  - **9.2.4.1.1.** Obtain the retention time for all single component compounds from the analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window for each analyte and surrogate by using the absolute retention time for each analyte and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. Apply the retention time window data in the table in Attachment 1 to its corresponding compound. Calculate absolute retention time windows for each analyte and surrogate on each chromatographic column and instrument.
  - **9.2.4.1.2.** New retention time windows must be established when whenever a chromatographic column is replaced or a new detector is installed .Whenever the observed retention time of each analyte and surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.
- 9.2.4.2. Initial Calibration Range: External standard calibration is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each analyte at each calibration concentration. The average response factor may be used for quantitation if the % RSD across the 5 point range is <20%. Alternatively, linear regression may be used if the correlation coefficient (r1) is ≥ 0.990 (note: if the linear regression model is used the curve must NOT be forced through the origin). Calibration is checked every 12 hours or after</p>

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every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single resonse pesticide standards.

**9.2.4.2.1.** Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

- **9.2.4.2.2.** If the % RSD across the 5 point range is <20% the calibration can be assumed to be linear and the average response factor can be used to calculate concentrations of target compounds in samples.
- 9.2.4.2.3. If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r<sup>1</sup> (Correlation Coefficient) value must be ≥ 0.990 for the calibration to be acceptable. Calibration is checked every 12 hours or after every twenty (20) samples, whichever comes first, by injecting a calibration verification standard for all single component pesticide standards.
- **9.2.4.2.4.** Chlordane and Toxaphene Calibration: Chlordane and Toxaphene are multiple response pesticides and are calibrated with a minimum of 5 points as required (i.e, within 12 hours of either analyte being detected in a sample). Three to eight peaks are used for calculation of response factors and the same criteria detailed above is applied to determine acceptability of calibration.
- **9.2.4.3.** Initial Calibration Verification (ICV): An ICV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2.4. The concentration of the ICV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.
- 9.2.4.4. Continuing Calibration Verification (CCV): An CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.3.. The concentration of the CCV must be within ±15%D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution,

perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

Step	Standards	Туре	Control Limit	Frequency
Method # 8081A		•	•	
GC System Performance Check	Endrin/DDT, 250 ppb	Performance check	15% breakdown	At beginning of 12 hour clock and after system maintenance
Initial Calibration	10, 50, 100, 250 and 500 ppb for single response, 1000 ppb for multi response	Average response factor or 1 <sup>st</sup> order linear regression	For average RF: <20%RSD all analytes. For linear regression: r ≥0.990	As required when ICV or CCV do not meet requirements
ICV	100 ppb	Average	± 15%D	Once after each initial calibration
CCV	100 ppb	Average	± 15%D	Every 12 hrs or 20 samples, whichever is more frequent

# 10. Procedure

# 10.1. Gas Chromatograph Operation

10.1.1. The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.

# 10.1.2. General Operating Conditions

- **10.1.2.1.** Injection System: A split/splitless injection port with electronic pressure control (EPC) is used. Thirty seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.
- **10.1.2.2.** The EPC is used in the ramp pressure mode. The ramp pressure program is as follows:

Initial Pressure	<u>InitialTime</u>	Rate	Final Pressure	<u>Hold</u>
12 psi	2.5 min	7 psi/min	4 psi	1.50 min
		5 psi/min	9 psi	1.40 min
		9 psi/min	13 psi	2.00 min

**10.1.2.3.** For pesticide analysis the normal operating conditions of the injection port are as follows:

Injection port Temperature: Column flow: Split vent flow: EPC: Detector temperature 250<sup>o</sup>C 12.3 ml/minute 5 ml/minute Pressure Ramp 330C

- **10.1.2.4.** In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. The plug of glass wool is located in the liner between the double goose neck.
- **10.1.2.5.** This liner/glass wool combination provides many functions. The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.
- **10.1.2.6.** The glass wool will be changed when changing the liner. The changing of the glass wool/liner is based upon the breakdown of an Endrin/DDT standard. This is covered in further detail in section 10.2.1.
- **10.1.2.7.** Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.
- 10.1.2.8. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.
- **10.1.2.9.** The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.

- **10.1.2.10.** Once the signal from both detectors has stabilized, it is time to reheat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.
- **10.1.2.11.** Oven: With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.
  - **10.1.2.11.1.** A standard oven program for pesticide analysis is employed for all columns as follows:

Initial Temp	Hold Time1	<b>Rate1</b>	<b>Temp1</b>
160°C	0.62 min	30°/min	244°C
Hold Time2	<b>Rate 2</b>	Final Temp	Final Time
2.5min	21°/min	315°C	3.0min

- **10.1.2.12.** Detectors: Detectors operate at 330°C and need to be supplied with 60 ml/min total flow. They are essentially maintenance free on a day-to-day basis. They are routinely baked out at 330°C to remove persistent contaminants. On occasion the detectors may be baked out at a higher temperature to remove contaminants with an extremely high boiling point (CAUTION: Do not exceed the maximum detector temperature of 380°C).
  - **10.1.2.12.1.** If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.
- **10.1.2.13.** Chemstation: The Chemstation is responsible for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.
- **10.1.2.14.** Target: Target is responsible for the processing of the data files. Calibrations, verification standards and samples are processed and reviewed using this database. All reports are also generated by Target.

#### 10.2. Analytical Sequence

- **10.2.1.** Setting Up for Analysis: The first operations to be performed when preparing for analysis are the calibration and performance checks.
- **10.2.2.** Performance and Check Standards: The instrument must first run the Endrin/DDT breakdown standard. This is to evaluate the performance of the injection port and column with regard to catalytic active sites. The breakdown must be less than 15% for both Endrin and DDT. If not, the performance check fails.
- 10.2.3. The instrument calibration for the 20 single response pesticides plus surrogates must next be check. This is accomplished by running the Pest Mix 100 ppb check standard. The actual concentration for each pesticide must be +/- 15 % of the expected concentration. Alternatively, the average concentration of <u>all</u> pesticides in the check standard must be ≤15%. If this criteria cannot be achieved for all pesticides and surrogates, the check standard fails.
- 10.2.4. The breakdown standard Endrin/DDT is acquired and measured before samples are analyzed and at the beginning of each 12 hour shift. If the breakdown check fails, then injection port/column maintenance is required. If a calibration check standard fails, then recalibration is necessary. <u>NOTE</u>: The 12 hour time clock for Pesticides commences with the injection of the first Pesticide Calibration Standard or Verification
- **10.2.5.** Calibration for Non-Pest Mix Pesticides: The instrument may be calibrated for quantitation of any semi-volatile, organochlorine pesticide. The pesticides most commonly calibrated for and not included in the Pest Mix are Chlordane and Toxaphene. Chlordane and Toxaphene are multiple response pesticides containing at least 3-8 primary peaks each. Both pesticides use area vs. concentration to generate a single point calibration using Target. The single point calibration check standard concentration is 1000ug/l for Chlordane and Toxaphene. The multiple peak responders calibration check standard should be analyzed only if the Pest Mix calibration ranges are being acquired.

Analytica	al Sequence			
1. Hexane	13. Hexane			
2. Instrument Blank	14. Instrument Blank			
3. Endrin/DDT Breakdown	15. Endrin/DDT Breakdown			
4. Pest Mix 1 (10 ppb)	16. Pest Mix 3 (100 ppb)CCV			
5. Pest Mix 2 (20 ppb)	17.20 or fewer samples or 12			
6. Pest Mix 3 (100 ppb)	hours			
7. Pest Mix 4 (250 ppb)	18. Instrument Blank			
8. Pest Mix 5 (500 ppb)	19. Endrin/DDT Breakdown			
9. Chlordane (1000 ppb)	20. Pest Mix 3 (100 ppb)CCV			
10. Toxaphene (1000 ppb)	21.20 or fewer samples or 12			
11. Pest ICV (100 ppb)	hours			
12. Resolution Check	22. Pest Mix 3 (100 ppb) CCV			

**10.2.6.** Analysis Sequence: The automation of GC runs is accomplished via the "SEQUENCE" macro of the Chemstation.

- **10.2.6.1.** After 20 samples or 12 hours of analysis, a Pesticide mix standard bracket must be analyzed. In order to continue with sample analysis these brackets must be within  $\pm$  15% of the expected concentrations. A second END/DDT breakdown must pass as well.
- **10.2.6.2.** The Sequence File: The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle.
- **10.2.6.3.** It is common practice to run the check standards, evaluate the instrument status, and then complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE soft-key of the SEQUENCE macro.
- **10.2.7.** Pesticide Report Printing: As previously mentioned, the Target Chromatography System will calculate the concentrations of 20 single response pesticides surrogates.

### 10.3. Dual Column Approach

- **10.3.1.** The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.
- **10.3.2.** The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.
- **10.3.3.** If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P\*.
- **10.3.4.** If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.
- **10.3.5.** If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.
- **10.3.6.** If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.

- **10.3.6.1.** An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.
- **10.3.7.** In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.
- 10.3.8. In summary, the flow chart in Attachment 2 presents a recommended rational approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

### 10.4. Extract Cleanups

- **10.4.1.** Cleanup methods are dictated by the original sample matrix and the parameters being determined.
- 10.4.2. Cleanup of all water samples, if needed, is performed using Florisil (TestAmerica Edison SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts, SW846 Method 3620B, most current revision) and TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B, most current revision). Blanks must also undergo cleanup following the same procedures as samples.
- 10.4.3. Cleanup of all soil samples is conducted using Florisil (TestAmerica Edison SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts, SW846 Method 3620B, most current revision) and, if needed, TBA sulfite (TestAmerica Edison SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B, most current revision). Blanks must also undergo cleanup following the same procedures as samples.
- **10.4.4.** Check Florisil prior to use to assure quantitative recovery of target analytes. Duplicate checks are required for each new lot and every three hundred

samples whichever is more frequent. (This is done in extractions and given to GC to analyze)

10.4.5. Check Florisil by spiking 1ml of the Pest Mix (Supelco Catalog No. 47426) midpoint and 0.5 ml of trichlorophenol (Protocol No. S-3640) onto the cartridge and concentrating to final volume of 1 ml. Inject 2 ul onto a capillary column, conducting the elution and analyzing the extract. Recovery is acceptable if all pesticides are recovered at 80 - 110% and the recovery of trichlorophenol is <5% and co-eluting interfering peaks are absent from the extract.</p>

### 10.5. Documentation

- **10.5.1.** Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, injector flow, signal A, signal B, analysts initials, and notes for any necessary repairs.
- **10.5.2.** After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor, method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

### 11.0. Calculations / Data Reduction

Refer to TestAmerica Edison SOP EDS-GEN-019 for Organic Calculations.

### 11.1. Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

MS % Recovery = <u>(spiked sample) - (unspiked sample)</u> x 100 spiked concentration

### 11.2. Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

### 11.3. Concentration

Concentration = mg/kg or L =  $C \times V \times D$ W Where: C = sample concentration in extract (ppm) V = Volume of extract (mL) D = Dilution Factor

W = Weight/Volume of sample aliquot extracted (grams or mLs)

**NOTE:** All dry weight corrections are made in LIMS at the time the final report is prepared.

### 12.0. <u>Method Performance</u>

### 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

### 12.2. Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

# 12.3. <u>Training Requirements</u>

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

# 13.0. Pollution Control

- **13.1.** Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.
- **13.2.** The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage.

### 14.0. Waste Management

- **14.1.** The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- **14.2.** The following waste streams are generated as a result of this analysis:
  - Expired Standards The vials are collected in a 1 gallon polyethylene bucket. These vials are then transferred to an open top 55 gallon steel or polyethylene waste drum. These drums are transported to a waste facility for proper disposal.
  - Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

Teris Profile Number: 50016652 Onyx Profile WIP Number: 282493

 Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624 Onyx Profile WIP Number: 545240

• Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710 Onyx Profile Number: (stabilization) 402535

# 15.0. <u>References / Cross-References</u>

- **15.1.** United States Environmental Protection Agency, "Method SW8000B: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.
- **15.2.** United States Environmental Protection Agency, "Method 8081A, Organochlorine Pesticide by Gas Chromatography", Test Methods for Evaluating Solid Wastes,

SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

- **15.3.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.4.** TestAmerica Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.
- **15.5.** TestAmerica Edison SOP No. ED-ORP-018: *Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.
- **15.6.** TestAmerica Edison SOP No. ED-ORP-016: Automated Soxhlet Extraction of Solid Samples Pesticides/PCBs, SW846 Method 3541, most current revision.
- **15.7.** TestAmerica Edison SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A, most current revision.
- **15.8.** TestAmerica Edison SOP No. ED-ORP-020: *Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.
- **15.9.** TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.
- **15.10.** TestAmerica Edison Work Instruction No. EDS-WI-017, *EPA Method 8081 Current Spike, RPD, Surrogate Limits,* most current revision.
- **15.11.** TestAmerica Edison Work Instruction No. EDS-WI-072, *SW846 Method 8081A, Current MDLs and Reporting Limits*, most current revision.
- **15.12.** TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
- **15.13.** TestAmerica Edison SOP No. ED-GEN-022, *Training*, most current revision.
- **15.14.** Test America Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*), most current revision.
- **15.15.** TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), most current revision.

### 16.0. Method Modifications:

#### Not applicable

### 17.0. Attachments

Attachment 1: Retention Time (RT) Windows For Single Analytes/Surrogates Attachment 2: Dual Column Reporting Flowchart

### 18.0. <u>Revision History</u>

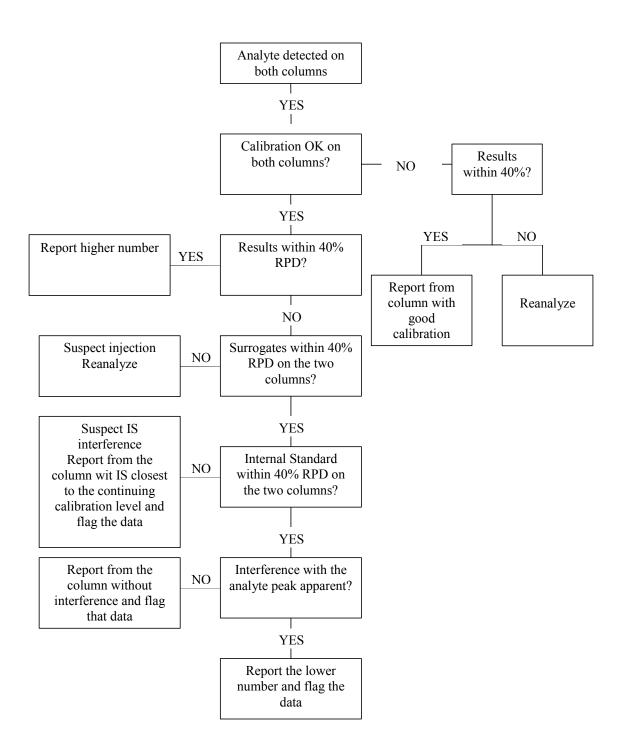
- Revision 6, dated June 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1. Added table detailing Reporting Limits by matrix.
  - Section 1.2 Added reference to Quality Assurance Manual for method modifications.
  - Section 2: Expanded to include references to applicable prep SOPs.
  - Section 3: revised to reference new location for definitions.
  - Section 4: Revised to include specific references to cleanup SOPs. Updated vendor and catalog number for standards used in Florisil check.
  - Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.1.3: added details of the solvent testing and approval program.
  - Section 7.2.1:Updated standards sources and catalog numbers.
  - Section 7.2.2: Added table detailing components found in the various standards mixes.
  - Section 7.2.2.1: Updated the instructions for preparation of the 5 point calibration range standards. Added a table with calibration standards prep details.
  - Sections 7.2.2.2 thru 7.2.2.9: Added details to standards prep text.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
  - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
  - Section 9.2: Expanded details of preparation, analysis, evaluation and corrective action for initial and continuing calibration and calibration verifications. Added a table summarizing Instrument QC Requirements.
  - Section 10.2.6: Updated Idealized Analytical Sequence table.
  - Section 10.3: Added a section detailing the Dual Column Approach for Method 8082.
  - Section 10.4: Added references for cleanup SOPs.
  - References: Expanded to include more specific SOP references
  - o Attachements: Added Attachment 1: Dual Column Approach
  - Section 18: Added this Revision History section

# ATTACHMENT 1

# Retention Time (RT) Windows For Single Analytes/Surrogates

Compound	RT Window (minutes)
alpha-BHC beta-BHC gamma-BHC (Lindane)	±0.05 ±0.05 ±0.05
delta-BHC	±0.05
Heptachlor	±0.05
Aldrin alpha-Chlordane	±0.05 ±0.07
gamma-Chlordane	±0.07
Heptachlor Epoxide	±0.07
Dieldrin	±0.07
Endrin Endrin aldehyde	±0.07 ±0.07
Endrin Ketone	±0.07
4,4'-DDD	±0.07
4,4'-DDE	±0.07
4,4'-DDT Endosulfan 1	±0.07 ±0.07
Endosulfan 11	±0.07
Endosulfan sulfate	±0.07
Methoxychlor	±0.07
Tetrachloro-m-xylene	±0.05
Decachlorobiphenyl	±0.10





**Edison** 



SOP No. ED-GC-04, Rev. 7 Effective Date: 01/12/2010 Page No.: 1 of 25

# Title: Analysis of Polychlorinated Biphenyls by Gas Chromatography using SW846 Method 8082

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# 1.0 Scope and Application

This method is used to quantify specific polychlorinated biphenyls (PCBs) as Aroclors (see Table 1 below) in extracts from aqueous, soil, sludge, leachate, wipe or oil matrices by direct injection dual capillary column gas chromatography. An electron capture detector (ECD) is employed for detection.

### 1.1 Analytes, Matrix(s), and Reporting Limits

Table 1 Polychlorinated Biphenyls			
Compound Name	CAS Registry No.		
Aroclor 1016	12674-11-2		
Aroclor 1221	11104-28-2		
Aroclor 1232	11141-16-5		
Aroclor 1242	53469-21-9		
Aroclor 1248	12672-29-6		
Aroclor 1254	11097-69-1		
Aroclor 1260	11096-82-5		
Aroclor 1262	37324-23-5		
Aroclor 1268	11100-14-4		

The specific analytes determined by this method are identified in Table 1.

The routine TestAmerica Edison reporting limits (RLs) by analyte and matrix are summarized in Table 2 (below).

Table 2 Reporting Limits by Matrix						
Parameter	Soil	Soil	Water	Leachate	Oil	Wipe
	Reporting Limits (ug/kg) LOW Level	Reporting Limits (ug/kg) MED Level	Reporting Limits (ug/L)	Reporting Limits (mg/L)	Reporting Limits (ug/kg)	Reporting Limits (ug/wipe)
Aroclor-1016	67	500	0.50	0.0050	1000	0.40
Aroclor-1221	67	500	0.50	0.0050	1000	0.40
Aroclor-1232	67	500	0.50	0.0050	1000	0.40
Aroclor-1242	67	500	0.50	0.0050	1000	0.40
Aroclor-1248	67	500	0.50	0.0050	1000	0.40
Aroclor-1254	67	500	0.50	0.0050	1000	0.40
Aroclor-1260	67	500	0.50	0.0050	1000	0.40
Aroclor-1262	67	500	0.50	0.0050	1000	
Aroclor-1268	67	500	0.50	0.0050	1000	

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work Request*) and

Section 20 (*Test Methods and Method Validation*) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

### 2.0 <u>Summary of Method</u>

- **2.1.** Samples undergo a preparation step prior to analysis by SW846 Method 8082. A measured volume or weight of sample (15 g for soil, 1 g for oil, 1000 ml for water, and 100 ml for TCLP) is extracted using the appropriate matrix-specific sample extraction technique (reference the applicable Organic Sample Prep SOPs listed below). The extract is exchanged into hexane and concentrated to a final volume between 5 and 20 ml depending upon the prep technique used.
  - 2.1.1. Aqueous and leachate samples are extracted at a neutral pH using either SW846 Method 3510C (SOP No. ED-ORP-014: Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C) or SW846 Method 3520C (SOP No. ED-ORP-003: Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C).
  - 2.1.2. Solid samples are extracted using SW846 Method 3550B: Sonication (SOP No. ED-ORP-018: Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B) or SW846 Method 3541 (SOP No. ED-ORP-016: Automated Soxhlet Extraction of Solid Samples Pesticides/PCBs, SW846 Method 3541).
  - **2.1.3.** Organic liquids are prepared using SW846 Method 3580A (SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A).
  - 2.1.4. Extract cleanup steps are employed as need depending on the nature of the matrix interferences encountered. Suggested cleanups include SW846 Method 3620B (SOP No. ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts), SW846 Method 3660B (SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts) and SW846 Method 3665A (SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A) for heavy organic interferences.
- **2.2.** After cleanup, the extract is analyzed by injecting a 2-uL sample into an Agilent Technologies gas chromatograph equipped with a dual wide-bore fused silica capillary columns and dual electron capture detectors (GC/ECD).
- **2.3.** Samples are analyzed only after all the necessary calibration and QC checks have been performed.
- **2.4.** Acquired data from sample analysis is manually reviewed. Secondary column confirmation of target compounds and quantitation are conducted by the analyst as required.

#### 3.0 <u>Definitions</u>

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 4.0 Interferences

- **4.1.** Interferences from phthalate esters introduced during sample preparation can pose major difficulties for PCB determinations.
  - **4.1.1.** Interferences from phthalate esters can be minimized by avoiding contact with any plastic materials and checking all solvents and reagents for phthalate contamination. Exhaustive cleanup of solvents, reagents and glassware may be required to eliminate background phthalate ester contamination.
- **4.2.** The presence of elemental sulfur will result in broad peaks that interfere with the detection of early-eluting PCBs. Sulfur contamination should be expected with sediment samples. Employ SW846 Method 3660B (SOP No. ED-ORP-021: The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts) for removal of sulfur.
- **4.3.** Co-eluting chlorophenols are eliminated by using SW846 Method 3620B (SOP No. *ED-ORP-020: Florisil Cleanup for Pesticide/PCB Sample Extracts*),
- **4.4.** Interferences from other organic compounds can effectively be removed using a sulfuric acid treatment, SW846 Method 3665A (*SOP No. ED-ORP-022, Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A*). This destructive technique can be employed only when the sample extract is being analyzed solely for PCBs (i.e., it is not to be used prior to analysis for pesticides).

### 5.0 Safety

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum

### 5.1. Specific Safety Concerns or Requirements

The gas chromatograph contains zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

There are areas of high voltage in the gas chromatograph. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

# 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Acetone	Flammable	1000 ppm- TWA	Inhalation of vapors irritates the respiratory tract. May cause coughing, dizziness, dullness, and headache.
Hexane	Flammable Irritant	500 ppm- TWA	Inhalation of vapors irritates the respiratory tract. Overexposure may cause lightheadedness, nausea, headache, and blurred vision. Vapors may cause irritation to the skin and eyes.
Methylene Chloride	Carcinogen Irritant	25 ppm- TWA 125 ppm- STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.
Sulfuric AcidCorrosive Oxidizer1 Mg/M3- TWAInhalation produces damaging effects on the mucous membranes and upper respiratory tract. Symptoms may include irritation of the nose and throat, and labored breathing. Symptoms of redness, pain, and severe burn can occur. Contact can cause blurred vision, redness, pain and severe tissue burns. Can cause blindness.			
1 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

# 6.0 Equipment and Supplies

# 6.1 Gas Chromatograph:

**6.1.1** Agilent Technologies (Avondale, PA) model 5890/6890 Gas Chromatograph (GC), equipped for simultaneous quantitation and confirmation columns using two separate detector channels on dual

megabore capillary columns that are suitable for the analysis of organochlorine pesticides and PCB's. All operations are as automated as possible with the equipment utilized.

- **6.1.2** Injection system: Sample injection is accomplished by a single auto injector. The auto injector is serviced by a robot arm that shuttles a single sample between the sample tray and the injector turret.
  - **6.1.2.1** The sample is injected into a split/splitless injection port equipped with electronic pressure control (EPC). The injection port is normally operated in splitless mode during injection. The EPC is operated in the constant pressure mode.
- **6.1.3** Liners: The injection port is each fitted with a replaceable, heavy-walled glass double gooseneck liner. The liner contains a plug of silanized glass wool approximately 1 cm in length. The glass wool is positioned in the liner between the double gooseneck. The liner is replaced on a regular maintenance schedule.
- **6.1.4** Oven and Columns: Temperature programmable gas chromatograph ovens are required, capable of integrated temperature control between 35°C and 350°C.
  - 6.1.4.1 Two dissimilar columns are used for analysis. A Restek StxCLPesticides, 30m x 0.53mm ID x 0.5um film thickness column (or equivalent) is used for sample quantitation. The secondary column is a Restek StxCLPesticides II, 30m x 0.53mm ID x 0.42um film thickness column (or equivalent).
- **6.1.5** Detectors: Sample detection is by electron capture. The GC is equipped with dual Electron Capture Detectors (ECD), one for each column.
  - **6.1.5.1** Each detector is supplemented with make-up gas to provide sufficient detector flow for maintaining the electron plasma. This is in addition to the gas exiting the column. The make-up gas (P-5) is fed from a supply other than the injection port.

# 6.2 Data System:

**6.2.1** The data systems consist of Agilent Technologies GC Chemstation Revision A.08.02 and Agilent Technologies Enviroquant Chemstation G1701AA Version A.03.00 upgraded to A.03.02 which is used for acquisition and Target software (Thru-Put Systems) using a Falcon integrator for data processing.

### 7. Reagents and Standards

# 7.1. <u>Reagents</u>

- 7.1.1. Gases: Ultra high purity (99.999%) Helium is used as the carrier and injection port purge gas. It is introduced to the GC at the injection port. Ultra high purity(99.999%) Argon (95%) / Methane (5%) (a.k.a. P-5 Mixture) is used as make-up gas. It is introduced to the GC via the make-up gas adapter at the end of the capillary column. They are supplied by M-G Industries (Valley Forge, PA). Both gases are supplied at tank pressures of 2000-2400 psig for a 300 cft tank. The tank pressure is regulated to an outlet pressure of 70 psig. Each tank is used until the tank pressure drops to less than 500 psig.
  - **7.1.1.1.** The gas streams are polished using three traps or filters before introduction to the G.C. The traps are as follows:
    - > Hydrocarbon trap
    - $\succ$  H<sub>2</sub>O (moisture) trap
    - $\triangleright$  O<sub>2</sub> scrubber
  - **7.1.1.2.** Both the moisture trap and the Oxygen scrubber are of the indicating type. They require either replacement or reconditioning upon color change of the active agents. Refer to the instructions for the individual traps to determine if it is still active. The hydrocarbon trap is a simple activated carbon trap. With high quality gas, it should last for an extended period of time (1-yr. minimum).
- **7.1.2.** Solvents used in the extraction, clean up procedures and dilutions include Hexane, Methylene Chloride, and Acetone that are exchanged to Hexane prior to analysis. All solvents must be pesticide quality or equivalent. Each lot of solvent is screened for contaminants before being used for analysis.

# 7.2. Standards

**7.2.1.** Standards are purchased as concentrated solutions. Standard compounds or mixtures for this analysis include an Aroclor 1016/1260 mix, Aroclor 1221, 1232, 1242,1248, 1254,1262, 1268 and the surrogate compound Decachlorobiphenyl (DCB) (packaged with the TCMX, a surrogate used in pesticide analysis).

NOTE: Two independent sources are used for quantitation standards and spiking standards

**7.2.1.1.** Most stock solutions are diluted (in volumetric glassware) to working concentration using hexane as the diluent.

Standard Name	Source	Concentration
TCMX/DCB Surrogate Calibration Mix	Supelco 48460	200 ug/ml
TCMX/DCB Surrogate Spike Mix	Supelco 861275	10 ug/ml
Aroclor 1016 Calibration Standard	Supelco 48097	1000 ug/ml

7.2.2. Standards mixes and sources: \*

Standard Name	Source	Concentration
Aroclor 1221 Calibration Standard	Supelco 48098	1000 ug/ml
Aroclor 1232 Calibration Standard	Supelco 44805	1000 ug/ml
Aroclor 1242 Calibration Standard	Supelco 44806	1000 ug/ml
Aroclor 1248 Calibration Standard	Supelco 44807	1000 ug/ml
Aroclor 1254 Calibration Standard	Supelco 44808	1000 ug/ml
Aroclor 1260 Calibration Standard	Supelco 44809	1000 ug/ml
Aroclor 1262 Calibration Standard	Supeclo 44810	1000 ug/ml
Aroclor 1268 Calibration Standard	Supelco 502146	1000 ug/ml
Aroclor 1660 Mix (Arolcors 1016 & 1260)	Supelco 861274	100 ug/ml
Aroclor 1016 Calibration Standard	Supelco 4S8097	1000 ug/ml
(Second Source)		
Aroclor 1260 Calibration Standard	Supelco 4S4809	1000 ug/ml
(Second Source)		

<sup>\*</sup>Suppliers with equivalent standards may be used.

7.2.3. Aroclor 1016/1260 & Surrogate Calibration Standard Solution Preparation

Five levels of calibration standards are prepared using the above referenced Aroclor 1660 Mix (Supelco – 861274) and TCMX/DCB Surrogate Calibration standard mix (Supelco 48460). They are prepared as follows:

Final Concentration of Aroclor 1016/1260 (Concentration of DCB)	Volume (ul) of Aroclor 1660 Mix (100 ug/ml)	Volume (ul) of TCMX/DCB Surrogate Calibration Mix (200 ug/ml)	Final Volume in hexane (ml)
100 ppb (25 ppb DCB)	10	12.5	100
500 ppb (50 ppb DCB)	50	25	100
1000 ppb (100 ppb DCB)	500	250	500
1500 ppb (150 ppb DCB)	150	75	100
2500 ppb (200 ppb DCB)	250	100	100

7.2.4. Surrogate Spiking Solution (soil and water)

The TCMX/DCB Surrogate Spike Mix (Supelco 861275) is used for spiking soils and waters as purchased from Supelco without additional dilution (10 ug/ml). This standard is purchased at 10 ug/ul in 50 ml volumes in Acetone.

### 7.2.5. Surrogate Spiking Solution (oil)

The TCMX/DCB Surrogate Spike Mix (Supelco 861275) must be further diluted prior to use in spiking oil/organic waste samples. To prepare a 0.5 ppm DCB & TCMX Surrogate Oil Spiking Solution dilute 2.5 ml of the 10 ug/ml solution to 50 ml with Acetone.

**7.2.6.** Aroclor 1016/1260 Spiking Solution (soil and water)

The Aroclor 1660 Mix (Supelco – 861274) is used spiking soils and waters as received from Supelco without further dilution. The standard is purchased at 100 ug/ul in 10 ml volumes in Acetone.

**7.2.7.** Aroclor 1016/1260 Spiking Solution (oil)

The Aroclor 1660 Mix (Supelco – 861274) must be further diluted prior to use in spiking oil/organic waste samples. To prepare a 5.0 ppm A1016/1260 Spiking Solution dilute 0.5 ml of the 100 ug/ml solution to 10 ml with Acetone.

**7.2.8.** Individual Aroclor Calibration Solutions (1221, 1232, 1242, 1248, 1254, 1262 & 1268)

A 1000 ppb calibration standard is prepared for each remaining Aroclor from the stock standards detailed in Section 7.2.1. 200ul of 1000 ug/ml individual Aroclor solution and 100ul of 200 ug/ml TCMX/DCB is diluted to 200ml with Acetone. The final concentration of surrogates is 100 ppb.

**7.2.9.** Aroclor 1016/1260 Initial Calibration Verification (ICV) Standard Solution Preparation

A mid-point Aroclor 1016/1260 ICV standard is prepared using the second source Aroclor 1016 Calibration Standard (Supelco 4S8097) and second source Aroclor 1260 Calibration Standard (Supelco 4S4809) detailed in Section 7.2.2. 500 ul of each 1000 ug/ml standard along with 250 ul of 200 ug/ml TCMX/DCB surrogate standard is diluted to 500 ml with acetone for a final ICV concentration of 1 ug/ml (1000 ppb).

# 8. Sample Collection, Preservation, Shipment and Storage

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml	Cool 4 <u>+</u> 2ºC	7 days to extraction; Analyze within 40 days of extraction	SW846
Soils	Glass, 2 or 4 oz	100 g	Cool 4 <u>+</u> 2ºC	14 days to extraction; Analyze within 40 days of extraction	SW846

**8.1.** Extracts must be stored under refrigeration in the dark and analyzed within 40 days of extraction.

### 9. Quality Control

**9.1.** <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) <sup>1</sup>	1 in 20 or fewer samples	Statistical Limits <sup>4</sup>
Matrix Spike (MS) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits 4
MS Duplicate (MSD) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits <sup>4</sup>
Surrogates	every sample <sup>3</sup>	Statistical Limits <sup>4</sup>

<sup>1</sup> LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

<sup>2</sup> The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

<sup>3</sup> Analytical and QC samples (MB, LCS, MS/MSD)

<sup>4</sup> Statistical control limits are updated annually and are updated into LIMS.

- **9.1.1. Method Blanks** are extracted with each sample batch on each day that samples are extracted. The analytical results for the method blank must fall below the reporting limit for each compound of interest. If a target compound is detected in the blank at a concentration higher than the reporting limit, first the extract is reanalyzed for confirmation. If results are still outside of limits the entire batch of samples extracted with the affected blank must be re-extracted and reanalyzed.
- **9.1.2.** Laboratory Control Sample (LCS): A Laboratory Control Sample (LCS) or blank spike must be extracted and analyzed for with each batch of 20 environmental samples. The LCS data is used to assess performance if the MS/MSD recoveries fall outside of established limits. The recoveries of the LCS must fall within lab generated acceptance criteria.. If the spiked sample recovery results fall outside the laboratory generated limits (refer to TestAmerica Edison Work Instruction No. EDS-WI-015, *EPA Method 8082 Current Spike, RPD, Surrogate Limits*), the LCS recovery is evaluated. If LCS recovery is within limits the poor sample recovery results are attributed to matrix interference. If the LCS recovery results are outside QC limits, first the extract is reanalyzed and if it is still outside the limits the entire QA batch must be re-extracted and reanalyzed.
- **9.1.3.** Matrix Spike/Matrix Spike Duplicate (MS/MSD): A Matrix Spike/Matrix Spike Duplicate (MS/MSD) pair is extracted and analyzed with every batch of 20 environmental samples. MS/MSD recoveries are evaluated against lab generated limits (refer to TestAmerica Edison Work Instruction No. EDS-WI-015, *EPA Method 8082 Current Spike, RPD, Surrogate Limits*). If the MS/MSD recovery limits fall outside of lab limits

the LCS recovery is evaluated and corrective action is taken as described in 9.1.2.

**9.1.4. Surrogate Standards:** All samples, blanks and QC samples are spiked with a 2 component surrogate standard mix containing TCMX & DCB (see Section 7.2). The percent recovery of the DCB surrogate standard is calculated and compared to lab generated limits (refer to TestAmerica Edison Work Instruction EDS-WI-015, *EPA Method 8082 Current Spike, RPD, Surrogate Limits*). If the DCB recovery is outside of acceptance limits the sample extract is reanalyzed to confirm. If the recoveries are still outside of limits the sample must be re-extracted and reanalyzed or the data flagged as "estimated concentration".

### 9.2. Instrument QC

### 9.2.1. Initial Calibration Range and Initial Calibration Verification (ICV)

- **9.2.1.1.** *Initial Calibration Range*: Aroclors 1016/1260 and the surrogate (DCB) are calibrated using a five-point calibration range using up to 8 Aroclor peaks per point. The reporting limit (RL) is equal to the low point of the calibration range. All other Aroclors are calibrated using a single point calibration up to 8 peaks at the anticipated midpoint of the calibration range. (Note: while up to eight major peaks are calibrated, as few as three peaks can be used in quantitation of sample results). Standards are prepared following the instructions in Section 7.2. The low
- **9.2.1.2.** *Initial Calibration Verification (ICV):* An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.9 and must be from a source separate from the standards used in the Initial Calibration Range.

### 9.2.2. Continuing Calibration Verification (CCV):

A mid-point Continuing Calibration Verification (CCV) standard must be analyzed every 12-hours or 20 samples (whichever is more frequent) and at the end of each analytical sequence. For multi-response pesticides a CCV must be analyzed within 12 hours of any multi-response pesticide detects.

### 9.2.3. Calibration Acceptance Summary

**9.2.3.1. Retention Time Windows:** Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. All gas chromatographs used for pesticides analysis at TestAmerica Edison are equipped with electronic pressure control (EPC). The use of EPC results in little retention time

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variability between analyses. Accordingly, retention time variability for the purpose of retention time window determination for standards analysis is extremely small. The default retention time window option must therefore be employed as follows to accommodate the excellent precision of EPC equipped systems.

- **9.2.3.1.1.** Establish the center of the retention time window for each characteristic peaks from each Aroclor and each surrogate by using the absolute retention time for each peak and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. (Note: The absolute retention times of the calibration standards are also checked against the retention time window established by the mid-point standard of the initial calibration.).
- **9.2.3.1.2.** Apply the retention time window data in Table 2 to each characteristic peak on each column.

Table 2 Retention Time (RT) Windows for Multicomponent Analytes/Surrogates		
Compound RT Window (minutes)		
Aroclors	<u>+</u> 0.07	
2,4,5,6-Tetrachloro-m-xylene	<u>+</u> 0.05	
Decachlorobiphenyl	<u>+</u> 0.10	

- **9.2.3.1.3.** Calculate absolute retention time windows for each peak and surrogate on each chromatographic column and instrument.
- **9.2.3.1.4.** Whenever the observed retention time of a surrogate is outside of the established retention time window, the analyst is advised to determine the cause and correct the problem before continuing analyses.
- **9.2.3.1.5.** New retention time window data must be generated whenever a chromatographic column is changed or a new detector is installed.
- **9.2.3.2. Initial Calibration Range**. External standard calibration is employed for this method. The response factor (defined as the ratio of the area to the standard concentration) is calculated for each characteristic peak in the Aroclor 1016/1260 standard at each calibration concentration. The percent relative standard deviation (% RSD) of the response factors for each individual peak in the Aroclor 1016/1260 mix on each column is then determined.

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**9.2.3.2.1.** Calculate the percent Relative Standard Deviation of the response factors for each compound at each level:

% RSD = (RF Standard Deviation/RF Mean) X 100

RF = Response Factor

- **9.2.3.2.2.** Linear Calibration: If the % RSD is less than 20% over its working range for at least 5 peaks in the Aroclor 1016/1260 mix, the linearity of the range is assumed for all Aroclors over the same analytical range. Each individual peak's response factor is used for quantitation of all the samples and verification standards. The average of the value calculated for each individual peak is used to report the concentration in the samples.
- **9.2.3.2.3.** Linear Calibration Using Least Squares Regression: If the % RSD is >20% for any given compound, a first order linear regression can be applied to the data to calculate the calibration curve and determine sample concentration. If this method is employed, the r squared value must be > 0.99 for the calibration to be acceptable

#### 9.2.3.3. Initial Calibration Verification (ICV):

- **9.2.3.3.1.** After the initial calibration range has been analyzed, an Initial Calibration Verification standard is analyzed on each column to verify the validity of the initial calibration range. The ICV standard must be from a standard lot independent of the standards used in the initial calibration range. The verification standard for PCBs is the mid-range Aroclor 1016/1260 standard at 1000ppb. (See Section 7.2.9 for details on the preparation of the ICV).
- **9.2.3.3.2.** At least five, pre-selected, characteristic peaks of Aroclor 1016/1260 plus surrogates in the ICV must be checked to verify the Initial Calibration Verification. The calculated concentration of the ICV must be within ±15%D of the expected concentration. Should the %D exceed 15%, the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the ICV. If the %D still exceeds 15% after a single ICV reinjection, a new Initial Calibration Range must be analyzed.

## 9.2.3.4. Continuing Calibration Verification (CCV):

**9.2.3.4.1.** A Continuing Calibration Verification (CCV) must be analyzed every 12 hours or after every 20 samples (whichever is more

frequent). The CCV will consist of a second source standard at or near the midpoint of the Initial Calibration Range analyzed at the frequency specified in Section 9.2.2. The calculated concentration of the CCV must be within  $\pm 15\%$ D of the expected concentration. Should the %D exceed 15% the analyst should take corrective action (check standard solution, perform instrument maintenance, etc.) and re-inject the CCV. If the %D still exceeds 15% after a single CCV reinjection, a new Initial Calibration Range must be analyzed.

The acceptance criteria for the Initial Calibration Range, the ICV and the CCV are detailed in the table below.

Step	Standards	Туре	Control Limit	Frequency
Method # 8082				
Initial Calibration Range	100, 500, 1000, 1500 and 2500 ppb for Aroclor 1016/1260, 1000 ppb for all remaining Aroclors	Average response factor or 1 <sup>st</sup> order linear regression	For average RF: <20%RSD all analytes. For linear regression: r <sup>2</sup> ≥0.990	Initially and as required when ICV or CCV do not meet requirements
ICV	1000 ppb	Average	± 15%D	Once after each initial calibration
CCV	1000 ppb	Average	± 15%D	Every 12 hrs or 20 samples, whichever is more frequent

#### 10. Procedure

## 10.1. Gas Chromatograph (GC) Operation

- **10.1.1.** The sequence of events for GC analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed. Then samples must be run on the instrument. Chromatograms and reports must be evaluated for content, integration and concentration. Re-runs and dilutions must be made based on the calibrations that were in effect at the time the sample was run. Lastly, a detailed analysis and calculations must be performed to determine the concentration of all the parameters for which the sample was analyzed.
- **10.1.2.** General Instrument Operating Conditions:
  - **10.1.2.1. Injection System**: A splitless injection port with electronic pressure control (EPC) is used. Seventy-five seconds after sample injection, the purge valve is turned on to facilitate the sweeping of any remaining residual solvent/sample from the injection port.

**10.1.2.2.** The EPC is used in the pressure Ramp mode. The ramp pressure program is as follows:

Initial Pressure	InitialTime	<u>Rate</u>	Final Pressure	<u>Hold</u>
25 psi	0.50 min	20psi/min	15 psi	2.00 min
		8 psi/min	12 psi	6.60 min
		10.0 min	16 psi	2.00 min

**10.1.2.3.** For PCB analysis the normal operating conditions of the injection port are as follows:

Injection Temperature:	250 <sup>0</sup> C
Injection Port Pressure:	25ml/min
Column flow:	33.2 ml/minute
Split vent flow:	60.0 ml/minute
Purge vent flow:	1.2 ml/minute
EPC:	Ramp pressure mode

- **10.1.2.4.** In addition to the EPC, the injection port is also equipped with a siltek-coated glass double goose neck liner that contains a 1 cm glass wool plug. This liner/glass wool combination provides many functions.
- **10.1.2.5.** The glass wool serves as a heat sink rapidly vaporizing solvent and samples resulting in higher response factors. The liner also protects the column head from accumulation of high boiling residuals and particulates.
- **10.1.2.6.** Regular maintenance is performed on the injection port. When the glass wool/liner is changed, the septa also must be changed. Injection port, oven and detector temperatures are lowered to ambient prior to "cracking" the system. This is so as to introduce a minimum of damaging oxygen molecules into the system.
  - 10.1.2.6.1. After the system has cooled, the old liner is removed. The injection port should be checked for particulate residues and cleaned as needed. A flashlight is usually required for this. After a new liner has been prepared it is placed into the injection port. A graphite seal is placed around the liner. The edges of the seal must be flat, not knife-edged, and free of nicks or burrs. If any of these conditions are not met, the graphite seal must be replaced as well. The graphite seal is critical to proper operation of the injection port. If in doubt, replace it.

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- **10.1.2.6.2.** The locking ring on the top of the injection port should be turned, with the wrench, about 1/8 turn past finger tight. The septum nut should never be tightened more than finger tight. After the injection port is reassembled, all column nuts inside the oven should be checked for leaks using Snoop (Supelco) or another suitable leak tester.
- **10.1.2.6.3.** The septa should be changed each time the injection port is opened. Another routine maintenance operation to improve column performance is the removal of the first 3 cm of the column.
- **10.1.2.6.4.** Once the signal from both detectors has stabilized, it is time to re-heat the zones. The zones should be heated in the order of detectors, oven and then injectors. This is to ensure that volatilized contaminants do not condense on the column or detector.
- **10.1.2.7. Oven:** With the megabore columns installed, temperature programming is employed to achieve higher resolution of compounds and shorter run times than could be accomplished using isothermal methods.
  - **10.1.2.7.1.** The oven program and pressure ramping for PCB analysis is employed for all columns as follows:

Initial Temp	Hold Time 1	<u>Rate1</u>	<u>Temp1</u>
164 <sup>0</sup> C	0.0min	12 <sup>0</sup> /min	234 <sup>0</sup> C
Hold Time2	Rate2	<u>FinalTemp</u>	<b>FinalTime</b>
2.4 min	40 <sup>0</sup> /min	325 <sup>0</sup> C	1.5min

- **10.1.2.8.** If the detectors are particularly contaminated, they must be sent to Agilent Technologies in Avondale, Pennsylvania for reconditioning. This should occur if the detector baseline is greater than 100 Hz. Detector reconditioning should be required at a maximum of biannually.
- **10.1.2.9.** Chemstation: The Chemstation is utilized for automation of runs and acquisition. The system is dedicated to a single GC and does not multitask. Therefore, data manipulation cannot be done while sample analysis is in progress. The data system acquires and stores all chromatographic data.
- **10.1.2.10.** Target: Target is utilized for the processing of the data files. Calibrations, verification standards and samples are processed

and reviewed using this database. All reports are also generated by Target.

#### 10.2. Analytical Sequence

- **10.2.1.** The instrument operating conditions should be set as detailed in Section 10.1.
- **10.2.2.** Once instruments conditions have been established, the Initial Calibration Range, calibration verifications and retention time windows must be established Section 9.2.
- **10.2.3.** The analytical sequence is established via the "SEQUENCE" macro of the Chemstation data system. The sequence file contains the name of Method file corresponding to the type of analysis to be performed, the range of samples to be run, and the number of injections per bottle. It is common practice to run the calibration and/or calibration verification standards, evaluate the instrument status, and, finally, (if all meet criteria) complete the Sample Table and Sequence File. If everything else is complete, the run is initiated using the START SEQUENCE softkey of the SEQUENCE macro
- **10.2.4.** An idealized analytical sequence including an Initial Calibration Range is presented in the table below.

Idealized Analytical Sequence with Initial Calibration Range				
Injection Number	Identification			
1	Hexane			
2	Instrument Blank			
3	Aroclor-1660 Level 1 Cal Std (100 ppb)			
4	Aroclor-1660 Level 2 Cal Std (500 ppb)			
5	Aroclor-1660 Level 3 Cal Std (1000ppb)			
6	Aroclor-1660 Level 4 Cal Std (1500 ppb)			
7	Aroclor-1660 Level 5 Cal Std (2500 ppb)			
8	Aroclor-1221 Level 3 Cal Std (1000 ppb)			
9	Aroclor-1232 Level 3 Cal Std (1000 ppb)			
10	Aroclor-1242 Level 3 Cal Std (1000 ppb)			
11	Aroclor-1248 Level 3 Cal Std (1000 ppb)			
12	Aroclor-1254 Level 3 Cal Std (1000 ppb)			
13	Aroclor-1262 Level 3 Cal Std (1000 ppb)			
14	Aroclor-1268 Level 3 Cal Std (1000 ppb)			
15	Initial Calibration Verification (Aroclor 1660)			
16	Hexane			
17	Continuing Calibration Verification			
	(Aroclor1660)			
18 thru 37	Client samples and QC Samples (MS/MSD,			
	LCS, Method Blank)			
19	Continuing Calibration Verification (Aroclor1660)			

Idealized Analytical Sequence with Initial Calibration Range		
Injection Number	Identification	
	(after 12 hours or 20 samples whichever more	
	frequent)	

\*May be up to 20 samples or 12 hours from the start of the calibration verification standards

- **10.2.5.** After 12 hours of analysis or 20 client samples (whichever is more frequent), an CCV standard mix must be analyzed. If this "bracket" standard fails the criteria listed in Section 9.2.4.3, all samples analyzed during the previous period must be re-analyzed bracketed with passing CCVs.
- **10.2.6.** PCB Data Reporting: The Target Chromatography System calculates the concentrations of the selected Aroclor Peaks. The reporting limit is based on the concentration of the lowest standard in the initial calibration, adjusted for the sample wt/vol, final volume, dilution factor and % moisture (No unqualified analytical results or non detects may be reported which correspond to an extract concentration less than the lowest standard in the calibration range).
  - **10.2.7.1.** The quantitative values for all confirmed analytes must agree within 40% between the primary column and the confirmation column.
  - **10.2.7.2.** If the quantitative values do not agree within 40%, the discrepancy must be noted in the report with a qualification

#### 10.3 Dual Column Approach

- **10.3.1.** The laboratory designates the rear column as the primary column and the front column as the secondary column. If the difference between the dual columns results in ≤40% RPD report the higher concentration.
- **10.3.2.** The values are calculated from the chromatographic peaks that fall within the daily retention time windows established from the most recent preceding calibration verification.
- **10.3.3.** If the calculated values are greater than 40% RPD of each other, report the lower concentration regardless of whether that result is from the primary or secondary column. Report the result with a flag of P\*.
- **10.3.4.** If the surrogates on one column are very different (>40% RPD) compared to the other column, this may be indicative of a bad injection or columnar blockage. The sample should be reanalyzed. If similar results are obtained following reanalysis, report the lower of the two numbers and describe the circumstances in the job summary and report case narrative.
- **10.3.5.** If one of the columns fails CCV criteria (but the CCV is between 15%-40% greater than expected value), the sample results shall be reported from the

compliant column. If the falls outside of acceptance criteria on the low side, reanalysis shall be performed.

- **10.3.6.** If the CCV on one of the columns is more than 40% different from the correct value, it can be assumed that there has been significant drift on that column. The sample shall be reanalyzed against an acceptable calibration.
  - **10.3.6.1.** An exception to this requirement would be if the CCV recovery on one column fails on the high side and >40% RPD but the associated samples were non-detect for all target analytes on both columns. In this case the non-detect results may be reported from the compliant column.
- **10.3.7.** In some cases where the sample chromatography is complex and has largely varying peaks concentrations, the chromatographic separation may not be sufficient on the 0.53mm ID columns. In this case a confirmatory analysis on an instrument with 0.32 ID columns may be required. The supplemental data produced using analysis on the 0.32mm ID 'microbore' column may minimize overlapping and baseline interference difficulties, and better resolves potential positive identifications. Use of this alternative chromatographic technique shall be noted in the job summary and report case narrative.
- **10.3.8.** In summary, the flow chart in Attachment 1 presents a recommended approach to selecting the better number to report for dual column data. It shall be noted that these recommendations may be overridden by project specific requirements and that they cannot cover all eventualities. The complexity of some data set will require the final decision to be made utilizing the judgment of experienced analysts. In some cases further cleanup steps to remove interferences may be appropriate.

#### 10.4 Extract Cleanup

- **10.4.1.** Cleanup methods are dictated by the original sample matrix and the parameters being determined.
- **10.4.2.** Cleanup of all water samples, if needed, is performed using Sulfuric Acid Permanganate and/or TBA sulfite. Refer to TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision and TestAmerica Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A, SW846 Method 3665A,* most current revision. Blanks must also undergo cleanup following the same procedures as samples..
- **10.4.3.** Cleanup of all soil samples is conducted using TBA sulfite and Sulfuric Acid Permanganate. Blanks must also undergo cleanup following the same procedures as samples.

**10.4.4.** Cleanup using Sulfuric Acid Permanganate effectively destroys the majority of organic material in the sample extract and should be used only when PCB is the only analysis to be performed on the sample extract.

## 10.5 Documentation

- **10.5.1** Before the analysis sequence is initiated the GC Performance and Repairs logbook must be filled out. It should contain the following information: date, injector temp, oven temp, detector temp, column A flow, column B flow, signal A, signal B, analysts initials, and notes for any necessary repairs.
- **10.5.2** After samples have been run, each standard and sample must be entered into the Instrument Run Log. The Instrument Run Log should contain the following information: run date, data file name, vial position, sample number, initial volume/weight, final volume, dilution factor method, job number, QA number, extraction date, lab prep batch, target batch signature of analyst at the bottom of each page, lot numbers for standards used, and result of run (O.K., dilution, non-inject, etc.).

## 11.0. Calculations / Data Reduction

**11.1.** Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100 spiked concentration

11.2. Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

**11.3.** On-column concentration each Aroclor in sample (ug/L)

= Aroclor area response in sample/Average response factor from initial calibration

**11.4** Final Arolcor concentrations in aqueous samples  $(ug/L) = \frac{(ug/L) (FV) (DF)}{IV \times 1000}$ 

Where:

ug/L	= on-column concentration in ug/L (see Section 11.3)
FV	= final sample volume in mls.
DF	= dilution factor

IV = initial sample volume in liters.

11.5	Final Arolcor co	oncentrations	in solid sar	nples (ug/kg) =	(ug/L) (FV) (DF)
------	------------------	---------------	--------------	-----------------	------------------

(IW) (DW)

Where:

- ug/L = on-column concentration in ug/L (see Section 11.3)
- FV = final sample volume in mls.
- DF = dilution factor
- IW = initial sample weight in grams
- DW = decimal dry weight

**NOTE:** All dry weight corrections are made in LIMS at the time the final report is prepared.

## 12.0. <u>Method Performance</u>

## 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

#### 12.2. <u>Demonstration of Capabilities</u>

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 12.3. <u>Training Requirements</u>

Refer to TestAmerica SOP No. ED-GEN-022, *Training*, for the laboratory's training program.

#### 13.0. Pollution Control

**13.1.** Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.

**13.2.** The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage

## 14.0. Waste Management

- 14.1. The U.S. Environmental Protection Agency requires that laboratory waste management practices conducted be consistent with all applicable rules and regulations. All waste will be disposed of in accordance with Federal, State and Local regulations. Where reasonably feasible, technological changes have been implemented to minimize the potential for pollution of the environment. Employees will abide by this method and the policies in section 13 of the Corporate Safety Manual for "Waste Management and Pollution Prevention."
- **14.2.** The following waste streams are generated as a result of this analysis:
  - Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

Teris Profile Number: 50016652 Onyx Profile WIP Number: 282493

 Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624 Onyx Profile WIP Number: 545240

• Soil Retain Samples - These samples if not flagged in the system for any hazardous constituents are transferred to poly-lined cubic yard boxes. These boxes when full are sent to stabilization or incineration. These materials are sent out as hazardous for lead and chromium

Teris Profile Number (incineration): 50016710 Onyx Profile Number: (stabilization) 402535

#### 15.0. <u>References / Cross-References</u>

**15.1.** United States Environmental Protection Agency, "Method SW8000B: Determinative Chromatographic Separations,", Test Methods for Evaluating Solid

Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

- **15.2.** United States Environmental Protection Agency, "Method 8082, Polychlorinated Biphenyls (PCBs) by Gas Chromatography", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.
- **15.3.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.4.** TestAmerica Edison SOP No. ED-ORP-014: *Extraction of Pesticides and PCBs in Water by Separatory Funnel, SW846 Method 3510C,* most current revision.
- **15.5.** TestAmerica Edison SOP No. ED-ORP-003: *Extraction of Semi-Volatile Organic Compounds in Water by Continuous Liquid-Liquid Extraction, SW846 Method 3520C,* most current revision.
- **15.6.** TestAmerica Edison SOP No. ED-ORP-018: *Extraction of Pesticides/PCBs in Soil Using Low-Level Extraction, SW846 Method 3550B,* most current revision.
- **15.7.** TestAmerica Edison SOP No. ED-ORP-016: Automated Soxhlet Extraction of Solid Samples Pesticides/PCBs, SW846 Method 3541, most current revision.
- **15.8.** TestAmerica Edison SOP No. ED-ORP-019: Waste Dilution for Pesticides and PCBs, SW846 Method 3580A, most current revision.
- **15.9.** TestAmerica Edison SOP No. ED-ORP-020: *Florisil Cleanup for Pesticide/PCB Sample Extracts*, SW846 Method 3620B, most current revision.
- **15.10.** TestAmerica Edison SOP No. ED-ORP-021: *The Removal of Elemental Sulfur from Pesticide/PCB Sample Extracts, SW846 Method 3660B,* most current revision.
- **15.11.** TestAmerica Edison SOP No. ED-ORP-022: *Sulfuric Acid Cleanup for PCB Extracts, SW846 Method 3665A, SW846 Method 3665A,* most current revision.
- **15.12.** TestAmerica Edison Work Instruction No. EDS-WI-015, *EPA Method 8082 Current Spike, RPD, Surrogate Limits,* most current revision.
- **15.13.** TestAmerica Edison SOP EDS-GEN-019, *Organic Calculations,* most current revision.
- **15.14.** TestAmerica Edison SOP No. ED-GEN-022, *Training,* most current revision.

#### 16.0. <u>Method Modifications:</u>

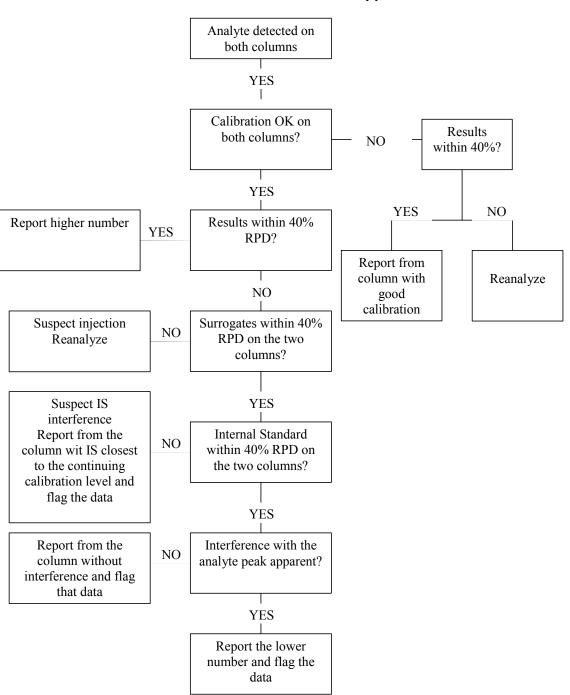
Not Applicable

## 17.0. Attachments

None

#### 18.0. <u>Revision History</u>

- Revision 7, dated 12 January 2009
  - Section 3.0: Updated to include the current location of the list of Definitions (Section 2 of the Quality Assurance Manual).
  - Section 11.0 and Section 15.0: Removed references to the now retired TestAmerica Edison SOP EDS-GEN-019, Organic Calculations.
  - Section 11.3 through 11.5: added more detail to calculations required for final reporting of Aroclor concentration in aqueous and solid samples.
- Revision 6, dated 26 May 2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1. Added Table 2, *Reporting Limits by Matrix*.
  - Section 1.1 Added reference to Quality Assurance Manual for method modifications.
  - Section 2: Expanded to include references to applicable prep SOPs.
  - Section 3: revised to reference new location for definitions.
  - Section 4: Revised to include specific references to cleanup SOPs.
  - Section 5: Revised to include most up to date corporate health and safety references and information.
  - Section 7.2.2: Added table detailing components found in the various standards mixes.
  - Section 7.2.3: Updated the instructions for preparation of the 5 point calibration range standards. Added a table with calibration standards prep details.
  - Sections 7.2.4 thru 7.2.9: Added details to standards prep text.
  - Section 8: Updated with additional details including a table outlining containers, preservation and holding times for waters and soils.
  - Section 9.1: Expanded QC sample preparation, analysis, evaluation and corrective action details.
  - Section 9.2: Expanded details of preparation, analysis, evaluation and corrective action for initial and continuing calibration and calibration verifications. Added a table summarizing Instrument QC Requirements.
  - Section 10.2.4: Updated Idealized Analytical Sequence table.
  - Section 10.3: Added a section detailing the Dual Column Approach for Method 8082.
  - Section 10.4: Added references for cleanup SOPs.
  - References: Expanded to include more specific SOP references
  - Attachments: Added Attachment 1: Dual Column Approach
  - Section 18: Added this Revision History section.



## Attachment 1 Dual Column Approach

### **TestAmerica Buffalo**



SOP No. BF-MV-005, Rev. 2 Effective Date: 03/30/2010 Page No.: 1 of 58 221T

# Title: Analytical Methods for the Analysis of GC/MS Volatiles [SW-846 Method 8260B]

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#### 1.0 Scope and Application

#### 1.1 <u>Analytes, Matrix(s), and Reporting Limits</u>

- **1.1.1** Methods 8260B -5 mL aqueous purge, 8260B 25mL aqueous purge, 8260B 5gr soil and 8260B medium level soil.
- **1.1.2** Applicable matrices include all aqueous samples, sediment, and soil.
- **1.1.3** The standard reporting limit (RL) is established at or above the low-level standard in the calibration curve. For a 5-ml purge volume, the RL for the majority of compounds is 1 ug/l.

On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in the Quality Assurance Manual.

#### 2.0 <u>Summary of Method</u>

- **2.1** This analytical method is utilized for the analysis of water, sediment and soil from hazardous waste sites for the organic compounds listed in Table 1.
- **2.2** The method includes sample preparation and analyses by purge and trap gas chromatograph/mass spectrometer (GC/MS). Method can be used for 5mL purge or 25mL purge (concentrations adjusted accordingly).
- **2.3** Volatile compounds are extracted from sample matrix by the purge and trap method. Analytes are desorbed onto a capillary column. An appropriate ramping temperature program is applied to maximize separation and achieve the correct resolution between the analytes. A mass spectrometer detector (MSD) interfaced to the gas chromatograph (GC) is utilized to detect analytes of interest.
- **2.4** Analytes eluted from the capillary column are introduced into the mass spectrometer via a direct connection. Identification of target analytes is accomplished by comparing their mass spectra with the electron impact spectra of authentic standards. Quantitation is accomplished by comparing the response of a major (quantitation) ion relative to an internal standard using a minimum of a five-point calibration curve.

#### 3.0 <u>Definitions</u>

- **3.1** <u>VBLK Volatile blank</u>: VBLK's are made from laboratory produced volatile free water. They are analyzed before samples to ensure a clean laboratory environment and analytical system.
- **3.2** <u>**IBLK Instrument Blank:**</u> IBLK's are made from laboratory produced volatile free water. They are analyzed after high level samples to verify that the system is clean and demonstrate the absence of carryover.
- **3.3** <u>LCS Laboratory Control Sample:</u> An LCS consists of a sample of volatile free water that is spiked with a group of target compounds representative of the method analytes. It is used to monitor the accuracy of the analytical process, independent of matrix effects.

- **3.4** <u>Surrogates (System Monitoring Compounds)</u>: Surrogates are organic compounds which are similar to the target analytes in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, VBLK, LCS and MS/MSD are spiked with surrogates.
- **3.5** <u>MS/MSD Matrix Spike/Matrix Spike Duplicate:</u> A Matrix Spike is an environmental sample which is spiked with a group of target compounds representative of the method analytes. A Matrix Spike Duplicate is a second aliquot of the same sample, which is spiked with the same target compounds. These samples are used to evaluate accuracy and precision in environmental samples.
- **3.6** <u>Batch:</u> A batch is a set of 20 samples using the same procedures within the same time period. Using this method each BFB analysis will start a new batch. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort must be made to keep the samples together.

## 4.0 Interferences

- **4.1** Airborne contamination may result from solvent vapors. VBLKs and IBLKs will be utilized to demonstrate a clean system and laboratory environment.
- **4.2** Some volatile compounds can permeate through a sample septum seal during storage or shipment. A weekly volatile holding blank is stored in all sample incubators to monitor contamination.
- **4.3** Contamination by carryover can occur whenever a sample with high concentrations of target compounds precedes a sample with low levels. The purging device, syringe and lines are flushed between every analysis to reduce carry over contamination. The trap is baked between each analysis.

## 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

#### 5.1 Specific Safety Concerns or Requirements

- **5.1.1** The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.
- **5.1.2** The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

**5.1.3** There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

## 5.2 Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material	Hazards	Exposure Limit (1)	Signs and symptoms of exposure
Methanol	Flammable Poison Irritant	200 ppm- TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.
1 – Exposure	e limit refers to	the OSHA reg	ulatory exposure limit.

## 6.0 Equipment and Supplies

## 6.1 <u>Instrumentation</u>

#### 6.1.1 Purge and trap devices

- Varian Archon Auto sampler
- Encon Concentrator
- O/I Analytical Auto sampler and Concentrator
- Centurion Auto sampler

#### 6.1.2 Trap Packing

- Vocarb 3000
  - Carbpack B
  - $\circ$  Carboxen 1000
  - o Carboxen 1001
- OI #10
  - o **Tenax**
  - Silica Gel
  - o cms
- Other traps may be used if the Quality Control criteria are met.

## 6.1.3 Gas Chromatograph/Mass Spectrometer (GC/MS) - GC: HP5890, MS:

- Gas chromatograph Column J&W Scientific DB-624 or Phenomenex ZB-624
- Internal diameter: 0.25mm or 0.18mm
- Length: 20m, 30m or 60m.
- Coating: Cyanopropylphenyl Methyl Silicone
- Film thickness: 1.0um or 3.0μm

## 6.1.4 Data System

- Computer with Chemstation enviroquant software
- Gas Chromatograph/Mass Spectrometer (GC/MS)-GC: HP6890 or HP7890, MS: Hewlett-Packard/Agilent 5973N or 5975.
- ProLab Resources software

## 6.1.5 Analytical Balance Mettler - Toledo Inc. Mettler AE160

#### 6.2 <u>Supplies</u>

- Syringes Hamilton Syringes size, 10ul, 25ul, 50ul, 100ul, 500ul, 1ml, 5ml, 10ml, 25ml
- Pasteur Pipettes disposable
- Vials and caps 2ml disposable
- Vials and caps 40ml disposable
- Volumetric flasks Pyrex 2ml, Pyrex 10ml, Pyrex 50ml, Pyrex 100ml
- pH paper wide range -.EM Science

#### 7.0 Reagents and Standards

- **7.1** <u>**Reagent Water**</u> For volatile analysis, the reagent water is volatile free and is prepared by passing water through a carbon trap.
- 7.2 <u>Methanol</u> Burdick & Jackson, purge and trap grade
- **7.3** <u>Stock Standards</u> Are purchased as certified standard mixtures. Traceability is documented following the procedures in the "Standards Traceability and Preparation Logbooks" SOP# AGP-STD-14. Individual compounds are prepared using reagent grade chemicals following the "Primary Standards Preparation" SOP# AMV-STD-25.
- 7.3.1 <u>Stock Target Compound Mix</u> Is composed of three different mixtures.
  - **7.3.1.1 <u>Gas Mix</u>** (See Table 7 for component list) is purchased at a concentration of 2000ug/ml.
  - **7.3.1.2 <u>54 Component</u>** Mix (See Table 8 for component list) is purchased at a concentration of 2000ug/ml.
  - **7.3.1.3 <u>8260+ Mix</u>** (See Table 9 for component list) is purchased and is composed of four separate mixtures.
    - 8260+ Mix #1 is purchased at a concentration of 1000ug/ml.
    - 8260+ Mix #2 is purchased at a concentration of 5000ug/ml.

- 8260+ Mix #3 is purchased at a concentration of 20000ug/ml.
- 8260+ Mix #4 is purchased at a concentration of 5000ug/ml.
- 7.3.2 <u>Stock Calibration Verification Mix</u> Is composed of two different mixtures.
  - **7.3.2.1** <u>The Second Source Mix</u> (See Table 10 for component list) is purchased at a concentration of 2000ug/ml.
  - **7.3.2.2** <u>The 8260+ Second Source Mix</u> (See Table 11 for component list) is purchased and is composed of two separate mixtures.
    - 8260+ Second Source Mix #1 is purchased at a concentration of 1000ug/ml.
    - 8260+ Second Source Mix #2 is purchased at a concentration of 5000ug/ml.
- **7.3.3** <u>Stock Internal Standard Solution</u> A mixture of 1,4-Dichlorobenzene-d4, Chlorobenzene-d5 and 1,4-Difluorobenzene in Methanol is purchased at a concentration of 2500ug/ml.
- **7.3.4** <u>Stock System Monitoring Solution</u> A mixture of Toluene-D8, 4-Bromofluorobenzene and 1,2-Dichloroethane-d4 in Methanol is purchased at a concentration of 2500ug/ml.
- **7.3.5** <u>Stock Matrix Spike Solution</u> A 5 component mixture of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene in Methanol is purchased at a concentration of 2500ug/ml.
- **7.3.6** <u>Stock BFB Solution</u> A solution of 4-Bromofluorobenzene in Methanol is at a concentration of 25000ug/ml.
- **7.4** <u>Secondary IS and System Monitoring Calibration Dilution Standards</u> these solutions are used for the manual injections required to prepare the initial calibration.
- **7.4.1** <u>Internal Standard Solution</u> 80ul of stock standard IS solution (2500ug/ml) is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.
- **7.4.2** System Monitoring Compound Solution 80ul of stock standard Surrogate solution (2500ug/ml) is added to approximately 1 ml of purge and trap methanol in a 2 ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol for a final concentration of 100ng/ml.
- **7.4.3** To calculate appropriate expiration dates, refer to "Standards Traceability and Preparation Logbooks".

#### 7.5 <u>Working Standards</u>

7.5.1 <u>Intermediate Calibration Solution</u> (Three individual mixtures)

7.5.1.1 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml

of purge and trap methanol in a 5ml Class A volumetric, and then brought up to a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.

- **7.5.1.2** 500ul of stock standard 54 Component Mix solution (2000ug/ml) is added to approximately 9ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.
- **7.5.1.3** 1000ul of each of the four stock standard 8260+ Mixes are added to approximately 5ml of purge and trap methanol in a 10ml Class A volumetric, and then brought up a final volume of 10ml with additional purge and trap grade methanol.
- **7.5.2** <u>Matrix Spike Solution</u> 100ul of stock standard 5 component solution (2500ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5 ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.
- **7.5.3** <u>A Full List Matrix Spike Standard</u> is made from stock Calibration Verification Standards and is composed of two mixes.
  - **7.5.3.1** 250ul of stock standard Gas Mix solution (2000ug/ml) is added to approximately 4 ml of purge and trap methanol in a 5ml Class A volumetric, and then brought up a final volume of 5ml with additional purge and trap grade methanol for a final concentration of 100ng/ul.
  - **7.5.3.2** 200ul of each of the two stock standard 8260+ Second Source Mixes are added to approximately 1ml of purge and trap methanol in a 2ml Class A volumetric, and then brought up a final volume of 2ml with additional purge and trap grade methanol.
- **7.5.4** <u>Working Internal Standard and System Monitoring Compound Solutions</u> for auto injection by instrument.
  - 7.5.4.1 Working Internal Standard Solution An Internal Standard Mixture is made from IS stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.
  - 7.5.4.2 Working System Monitoring Calibration Solution A System Monitoring Compounds Mixture is made from Surrogate stock standard (2500ug/ml). For water analysis a concentration between 20 and 30ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 25ug/L in the sample. For low level soil analysis a concentration between 45 and 55ng/ul is prepared, depending on sample loop size of the auto sampler, to produce a final concentration of 50ug/Kg in the sample.
- **7.5.5** <u>**Tuning Mixture**</u> 4ul of stock solution 4-Bomofluorobenzene (BFB) tuning mixture is added to approximately 1 ml of purge and trap grade methanol in a 2 ml Class A

volumetric, and then brought up to final volume of 2 ml with additional purge and trap grade methanol for a final concentration of 50ng/ul.

#### 7.5.6 Working Initial Calibration Standards

#### 7.5.6.1 Water: 25 mL

- **7.5.6.1.1** 20ul, 10ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 15ul, 5ul and 0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of three 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 40, 20 and 10 ug/L standards respectively.
- **7.5.6.1.2** 4ul and 1ul each of Intermediate Calibration Solution (7.5.1) and System Monitoring Compound Solution (7.4.2) are added to reagent water in 100 ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 4ug/L and 1ug/L standards respectively.
- **7.5.6.1.3** Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

#### 7.5.6.2 Water: 5 mL (5 point curve)

- **7.5.6.2.1** 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of four 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 100, 50, 25 and 10 ug/L standards respectively.
- **7.5.6.2.2** 1ul of each Intermediate Calibration Solution (7.5.1) and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in a 100ml volumetric flask. The flask is brought to volume with reagent water to prepare the 1ug/L standard.
- **7.5.6.2.3** The standard is then transferred into a 40ml vial and loaded onto the auto sampler.

#### 7.5.6.3 Water: 5 mL (6 point curve)

- **7.5.6.3.1** 50ul, 25ul 12.5ul and 5ul each of Intermediate Calibration Solution (7.5.1) and 37.5ul, 12.5ul, 0ul and 5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of four 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 100, 50, 25 and 10 ug/L standards respectively.
- **7.5.6.3.2** 5ul and 1ul of each Intermediate Calibration Solution (7.5.1) and 5.0ul and 1.0ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of two 100ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 5.0 and 1.0ug/L standards respectively.

**7.5.6.3.3** Each standard is then transferred into a 40ml vial and loaded onto the auto sampler.

## 7.5.6.4 Soil: (5 point curve)

- **7.5.6.4.1** 100ul, 50ul, 25ul, 10ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of five 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20 and 5 ug/kg, standards respectively.
- **7.5.6.4.2** 5 ml of each standard is then transferred into five individual 40ml vials and loaded onto the auto sampler.

## 7.5.6.5 Soil: (6 point curve)

- **7.5.6.5.1** 100ul, 50ul, 25ul, 10ul, 5ul and 2.5ul each of Intermediate Calibration Solution (7.5.1) and 75ul, 25ul, 0ul, 10ul, 5ul and 2.5ul of System Monitoring Compound Solution (7.4.2) is added to reagent water in each of six 50ml volumetric flasks. The flasks are brought to volume with reagent water to prepare the 200, 100, 50, 20, 10 and 5 ug/kg, standards respectively.
- **7.5.6.5.2** 5 ml of each standard is then transferred into six individual 40ml vials and loaded onto the auto sampler.

#### 7.5.7 Continuing Calibration Standard

#### 7.5.7.1 Water: 25 ml

**7.5.7.1.1** 5ul of stock target compound mix is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 10ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

#### 7.5.7.2 Water: 5 ml

**7.5.7.2.1** 12.5ul of stock target compound mix is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 25ppb. Pour the standard into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

## 7.5.7.3 <u>Soil:</u>

**7.5.7.3.1** 25ul of stock target compound is added to approximately 49mls of reagent water in a 50ml volumetric flask. The volumetric is brought to a final volume of 50ml to make a final concentration of 50ppb. Take 5ml and transfer it into a 40ml vial. The auto sampler adds the internal standard and system monitoring compounds.

#### 7.6 <u>Storage of Standards</u>

- **7.6.1** Stock standards are stored in flame sealed ampoules at  $22^{\circ}$  C to  $-20^{\circ}$  C according to the vendor's specifications.
- **7.6.2** Secondary dilution standards are stored in Teflon-sealed crimp cap vials at  $< 0^{\circ}$  C.
- **7.6.3** Aqueous standards are stored in Teflon-sealed vials at  $4^{\circ}$  C  $\pm 2^{\circ}$  C.

#### 8.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

- **8.1** Samples are collected in 40 mL vials with caps and septa, preserved to a pH < 2 with Hydrochloric Acid and stored at 4+2 degrees C until time of analysis.
- **8.2** Holding time for unpreserved samples is 7 days from sample date. For preserved samples the holding time is 14 days from sample date.
- **8.3** For some clients, regulatory agencies or QAPPS, the specified holding times may be different than those described in 8.2. In those cases, consult the specific Protocol/Method/QAPP or Project Manager for holding time details.

#### 8.4 <u>Sample Storage</u>

- Volatile samples are stored at  $4\pm 2^{\circ}$ C from the time of collection until analysis.
- Volatile samples are stored together in refrigerators specifically designated for volatiles only.
- Storage blanks are stored with samples until analysis.
- Samples and extracts are stored separately.
- Volatile samples and standards are stored separately.

#### 8.5 Preparation Of MS/MSD Samples

- 8.5.1 <u>Water Samples</u>: 40ml vial is spiked with 8ul of 50ng/ul or 4ul of 100ng/ul matrix spike standard for 25ml purge and 22ul of 50ng/ul or 11ul of 100ng/ul for the 5ml purge. This corresponds to a final concentration in the samples of 10ug/L and 25ug/L respectively. Analysis proceeds according to procedures described for water analysis.
- **8.5.2** <u>Low Level Soil/Sediment Samples</u>: 5ul of 50ng/ul or 2.5ul of 100ng/ul of matrix spiking solution is added to a 5g aliquot of sample. This corresponds to a final concentration in the samples of 50 ug/kg. Analysis proceeds according to procedures described for low-level soil/sediment samples.
- **8.5.3** <u>Medium Level Soil/Sediment Samples</u>: 1ml of methanol containing the soil spiking solution is combined with 50 mL of water. Sample analysis proceeds according to procedures described for medium level soil/sediment samples.

## 9.0 Quality Control

#### 9.1 Blank Analysis

- **9.1.1** <u>Method Blank:</u> A method blank consisting of a clean reference matrix (reagent water or purified quartz sand) must be analyzed prior to the analysis of samples but following any standard analysis.
  - Target compounds detected in a method blank must fall below the reporting limit, unless specified in client QAPP.
  - If internal standard or systems monitoring compound recoveries are not met, the method blank must be reanalyzed before the analysis of samples.
- **9.1.2** <u>Storage (Holding) Blank:</u> A weekly holding blank is analyzed to determine if cross contamination occurs within the volatile holding area. The results are reviewed by the quality assurance department and deemed acceptable or not acceptable. Corrective action, if necessary, will be taken.
- **9.1.3 Instrument Blank:** An instrument blank consisting of a clean reference matrix analyzed after the analysis of samples containing target compounds which exceed the calibration range. Multiple instrument blanks are shot until the instrument blank meets the criteria for method blanks.
- **9.2** <u>Matrix Spike Blank (MSB/LCS)</u> An aliquot of clean reference material spiked with the matrix spiking solution is analyzed with each analytical batch.
- **9.2.1** If a compliant Second Source Calibration Verification (SCV) has already been analyzed, then standards from the primary (CCV) source may be used. The solution is spiked at a concentration of 10ug/L for 25ml analysis, 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.
- **9.2.2** Alternatively, a standard that is purchased from an alternate vender (or where not available from a second vendor an alternate lot will be used) from the continuing (CCV) standard may be used. The solution is spiked at a concentration of 10ug/L for 25ml analysis, 25ug/L for 5ml analysis and 50ug/Kg for soil analysis.
- **9.2.3** The MSB/LCS must fall within internally derived statistical control limits or where applicable the limits specified by a project QAPP.
- **9.2.4** Analytes that have been identified as a Poor Performing Compounds (Table 5) will be considered compliant as long as their percent recovery exceeds 10%.
- **9.2.5** Routine compounds included in the MSB/LCS are:

1,1-Dichloroethene; Chlorobenzene; Toluene; Benzene; Trichloroethene

**9.2.6** When required, the MSB/LCS a 'full-compound' spike will be prepared and the MSB/LCS will be spiked with all compounds of interest. Due to the potentially large number of target compounds for method 8260B, it is possible that a few of the spiking compound could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that

compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable.

- **9.2.7** If the results of sample matrix spikes fall outside of the quality control range due to matrix, the MSB is used to verify that the laboratory can perform a spike on a clean matrix.
- **9.3** <u>Matrix Spike And Matrix Spike Duplicate Analysis</u> A matrix spike and matrix spike duplicate consisting of an actual field sample which has been spiked with the matrix spiking solution.
- **9.3.1** Matrix spike and matrix spike duplicate analysis will not be performed on rinsates or field/trip blanks.
- **9.3.2** If a sample has not been designated for MS/MSD analysis by the client, a sample will be selected at the analyst's discretion. MS/MSD analysis will be performed at a minimum of every 20 samples.
- **9.3.3** If insufficient sample was received for a designated MS/MSD the client will be contacted with the laboratories in-house designated sample for MS/MSD analysis. If no MS/MSD is required, the instance will be documented in the SDG narrative.
- **9.3.4** If medium level analysis is required on the client designated sample, the laboratory analyst will choose a low level sample on which to perform the quality control analysis. Medium level QC will also be performed.

#### 9.4 Data Assessment & Acceptance Criteria for QC Measures

#### 9.4.1 <u>Technical Acceptance Criteria For Initial Calibration</u>

**9.4.1.1** <u>SPCCs</u> (System performance check compounds) are compounds used to check compound instability degradation. The following average minimum average response factors must be met before the curve can be used.

Chloromethane	0.10
1,1-Dichloroethane	0.10
Bromoform	0.10
Chlorobenzene	0.30
1,1,2,2-Tetrachloroethane	0.30

**9.4.1.2 <u>CCCs</u>** (Calibration Check Compounds) evaluate the calibration based on the integrity of the system. The % RSD for the CCCs MUST be equal or less than 30%. The CCCs are:

Vinyl chloride
1,1-Dichloroethene
Chloroform
1,2-Dichloropropane
Toluene
Ethyl benzene

If the % RSD of any of the target analytes is 15% or less, the average response factor is assumed constant and the average response factor may be used for quantitation.

OR

If the % RSD of a target analyte is greater than 15%, linear regression or quadratic regression (forcing through the origin is advisable in both these models, to increase the accuracy at the low end of the calibration curve) may be used providing the coefficient of determination is greater than or equal to 0.99. If quadratic regression is used, a minimum of 6 calibration points must to be analyzed.

- **9.4.1.3** Non-standard analytes are sometimes requested for analysis by this method. For these analytes it is acceptable to analyze a single point standard at the reporting limit with each continuing calibration rather than a five point calibration. If the analyte is not detected in the associated samples a non-detect will be reported and no further action is required. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.
- **9.4.1.4 <u>Second Source Calibration Verification</u>** To verify the accuracy of the initial calibration, a standard is obtained from a source different from the Calibration Standards. This is also referred to as an SCV.
  - **9.4.1.4.1** Following the analysis of an acceptable initial calibration curve, an aliquot of this independent standard is analyzed at the CCV level.
  - **9.4.1.4.2** Recoveries of all compounds shall fall within ±30% of the expected values. However, the recoveries of up to 40% are allowable for up to four compounds.

## 9.4.2 <u>Technical Acceptance Criteria For Continuing Calibration</u>

- **9.4.2.1** <u>SPCCs</u> A system performance check is made daily or during every 12 hour analytical shift. Each compound must meet its minimum response factor (see Initial Calibration Criteria).
- **9.4.2.2** <u>CCCs</u> Used to check the validity of the initial calibration. The % Difference for each CCC shall be less than or equal to 20% from the initial calibration for the continuing calibration to be valid. All non-CCC target compounds must be less than 50% difference (or Drift) with allowance for up to six target analytes to be greater than 50%.
- **9.4.2.3** Internal Standard Retention Time The retention times for all internal standards must be evaluated to make sure that they are no more than 30 seconds from that of the midpoint of the initial calibration. If the retention time shift is greater than 30 seconds, the system must be inspected for malfunctions and maintenance must be performed, as required.
- 9.4.2.4 Internal Standard Response The EICP area for all internal standards must be

evaluated to make sure that they have not change by a factor greater than two (-50% to +100%) from that of the midpoint of the initial calibration. If the response exceeds these limits, the system must be inspected for malfunctions and maintenance must be performed, as required.

## 9.4.3 <u>Technical Acceptance Criteria of Quality Control Samples</u>

Samples, blanks, matrix spikes, and matrix spike duplicates must meet internal standard and system monitoring compound recovery limits. Where the Internal Standard recovery limit equals sample internal standard characteristic ion area (EICP) divided by the CCV internal standard characteristic ion area (EICP), multiplied by 100.

## 9.5 Corrective Action for Out-of-Control Data

## 9.5.1 Corrective Actions For MS/MSD

- 9.5.1.1 If the recoveries of the internal standards and system monitoring compounds do not agree with the unspiked sample (i.e. the sample recoveries were within control limits and MS/MSD recoveries were outside of control limits) the MS/MSD will be evaluated. The analyst will use their technical judgment to determine if the non-conformance is due to sample matrix or laboratory error. If it is determined that the QC failure was due to laboratory error, then reanalysis will occur.
- **9.5.1.2** If the recoveries of the internal standards and system monitoring compounds agree with the unspiked sample (i.e. both the sample and MS/MSD recoveries were outside of control limits) re-analysis is not required. The instance will be documented in the SDG narrative.
- **9.5.1.3** The laboratory on an annual basis establishes limits for the matrix spiking compounds. If the concentrations determined in the MS/MSD do not meet the control limits, no corrective action is necessary as long as the MSB/LCS was within control limits. The instance will be documented in the job narrative.

#### 9.5.2 Corrective Actions For Initial Calibration

- **9.5.2.1** If technical acceptance criteria cannot be met, it may be necessary to re-analyze the initial calibration. If after re-analysis, the criteria have not been met, it may be necessary to inspect the GC/MS system for possible problems.
- 9.5.2.2 Corrective actions may require one or several of the following procedures:
  - Open new/remake standard mixes
  - The ion source may be cleaned
  - The column may be cut at the injection port end
  - Change the purge trap on the purge and trap unit
  - Correct purge gas flow to optimize response
  - The column may be baked out

- The purge trap may be baked out
- The column may be replaced

## 9.5.3 <u>Corrective Actions for Failure to Meet the Continuing Calibration Acceptance</u> <u>Criteria</u>

- **9.5.3.1** If the technical acceptance criteria given above are not met, it may be necessary to reanalyze the continuing calibration check. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.
- **9.5.3.2** A single point standard at the reporting limit may be analyzed before the analysis of any samples. If the analyte is not detected in the associated samples a non-detect will be reported and a comment in the case narrative will be made. If the analyte is detected in any of the samples, a five point calibration will be analyzed and the samples with a positive detection will be re-analyzed against this compliant curve.
- **9.5.3.3** Other Corrective actions may be taken. The following details possible corrective actions:
  - Open new/remake standard mixes
  - The ion source may be cleaned
  - The column may be cut at the injection port end
  - The trap on the purge and trap unit may be replaced
  - The purge gas flow may be adjusted
  - The column may be baked out
  - The trap may be baked out
  - The column may be replaced

#### 9.5.4 Corrective Actions For Samples

- **9.5.4.1** If the internal standard or system monitoring criteria are not met, the sample must be re-analyzed to insure that it was not an internal problem that affected recoveries. If, after re-analysis, recoveries are outside of control limits, a matrix effect can be assumed.
- **9.5.4.2** When dilutions are performed, target compound concentration must fall within the upper range of the initial calibration. If any target compound exceeds the calibration range, the sample would require dilution. The sample immediately following a sample with target compounds above the calibration range must be monitored to insure that there is no carryover present. If there is a possibility of carryover, that sample must be re-analyzed.
- **9.5.4.3** If matrix effects exist, and both analyses exhibit recoveries outside of control limits, both analyses will be reported and documented in the job narrative.
- 9.5.4.4 lf, after re-analysis, recovery criteria are met, only the second analyses will be

reported. If the second analyses occur outside of the contract required holding time, both analyses will be reported in that instance.

**9.5.4.5** In the case of a matrix spike or matrix spike duplicate, these samples should only be reanalyzed if an error was identified in preparation or analysis of the sample. Failures will be documented in the SDG narrative.

## 9.5.5 <u>Corrective Actions for Failure to Meet the Laboratory Control Sample (Matrix Spike</u> <u>Blank) Acceptance Criteria</u>

- **9.5.5.1** The laboratory on an annual basis establishes limits for the matrix spiking compounds. The LCS must fall within these control limits. When required, the LCS will be spiked with all compounds of interest, otherwise spiked to include a minimum of 1,1-dichloroethene, trichloroethene, chlorobenzene, toluene, and benzene. Due to the potentially large number of target compounds for method 8260B, it is possible that a few of the spiking compounds could fall outside limits in the MSB/LCS. If a compound falls outside limits biased high and that compound is not found in the samples, a comment will be made in the case narrative and the data will be found to be acceptable
- **9.5.5.2** If the technical acceptance criteria are not met, it may be necessary to re-analyze the matrix spike blank. If, after re-analysis, the given criterion has not been met, it may be necessary to re-analyze the initial calibration.
- **9.5.5.3** Other Corrective actions may be taken. The following details possible corrective actions:
  - Open new/remake standard mixes
  - The ion source may be cleaned
  - The column may be cut at the injection port end
  - The trap on the purge and trap unit may be replaced
  - The purge gas flow may be adjusted
  - The column may be baked out
  - The trap may be baked out
  - The column may be replaced

#### 9.5.6 Corrective Actions for Failure to Meet the Method Blank (VBLK) Acceptance Criteria

- **9.5.6.1** If the technical acceptance criteria are not met, it may be necessary to re-analyze the associated samples.
- **9.5.6.2** If the analyte is a common laboratory contaminant (Methylene Chloride, Acetone, 2-Butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit.
- 9.5.6.3 If the target analyte is not greater than the reporting limit in the samples with the non-

compliant blank, the data may be reported with the analyte qualified.

**9.5.6.4** If surrogate recoveries are not acceptable, the data may be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination.

## 9.5.7 <u>Contingencies for Handling Out-of-Control or Unacceptable Data</u>

- Inform project manager for client input and fill out job exception report.
- Rerun samples to confirm results.
- Resample if client or project manager requests.

### 10.0 Procedure

## 10.1 <u>Calibration & Standardization</u>

#### 10.1.1 Instrument Tuning and Performance Check:

The GC/MS system is calibrated using Perflurotributylamine (PFTBA) according to the recommended tuning conditions suggested by the vendor.

An instrument performance check of Bromofluorobenzene (BFB) is analyzed at the beginning of each 12-hour analysis period.

The analysis of the instrument performance check is performed using the following procedure:

- 1ul of a 50ng/ul solution is directly injected, resulting in a 50ng injection of BFB into the GC/MS.
- A blank containing 50 ng BFB is purged.

#### 10.1.2 The mass spectrum of BFB is acquired using the following procedure:

- The apex scan, one scan immediately preceding the apex and one scan immediately following the apex are averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB.
- A scan across the peak at one half the peak height may be averaged. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak.
- A single scan of the peak may also be used for the evaluation of the tune. The spectrum is background subtracted using a single scan no more than 20 scans prior to the elution of BFB. Background correction cannot include any part of the target peak
- The mass spectrum of BFB must pass the technical acceptance criteria given in Table 2.

#### 10.1.3 Initial Calibration (ICAL):

The instrument performance check must meet the technical acceptance criteria prior to the

analysis of an initial curve or samples. The GC/MS system is calibrated using a minimum of five levels of concentrations. All compounds of interest are included. (See section 9.4 for initial calibration acceptance criteria.)

Solutions containing target compounds and system monitoring compounds are analyzed at the following concentrations:

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD005*	MeOH	100ng/ul	5	100	5
VSTD010	MeOH	100ng/ul	5	50	10
VSTD025	MeOH	100ng/ul	12.5	50	25
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100

#### 5 gram (soil) Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. (ug/kg)
VSTD005	MeOH	100ng/ul	2.5	50	5
VSTD010*	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD050	MeOH	100ng/ul	25	50	50
VSTD100	MeOH	100ng/ul	50	50	100
VSTD200	MeOH	100ng/ul	100	50	200

## 25 ml Purge Analysis

Standard	Solvent	Working Standard Conc.	Amount Added (ul)	Final Vol. (mL)	Final Conc. Water (ug/L)
VSTD001	MeOH	100ng/ul	1	100	1
VSTD004	MeOH	100ng/ul	4	100	4
VSTD010	MeOH	100ng/ul	5	50	10
VSTD020	MeOH	100ng/ul	10	50	20
VSTD040	MeOH	100ng/ul	20	50	40

\* optional 6<sup>th</sup> point for the initial calibration

## 10.1.4 Continuing Calibration Verification (CCV):

Every 12 hours of sample analysis the laboratory must demonstrate that the instrument has

drifted or changed minimally by performing an instrument performance check and continuing calibration verification. (See section 9.4 for continuing calibration acceptance criteria.)

#### 10.2 <u>Before Analysis</u>

- **10.2.1** Once initial calibration criteria has been met, and prior to analyzing samples and required blanks, Each GC/MS system must be routinely checked by analyzing a Continuing Calibration Verification (CCV) standard containing all compounds (including internal standards and system monitoring compounds) at a concentration of 25ug/L for 5ml analysis, 10ug/L for 25ml analysis or 50ug/Kg for soil.
- **10.2.2** If time remains after initial calibration criteria have been met, it may not be necessary to perform a CCV. The 25 ug/L (10ug/L for 25ml or 50ug/Kg for soil) standard may be evaluated against the new initial curve and used as the CCV.
- **10.2.3** If there is no time remaining in the 12-hour period, the instrument performance check (BFB) must be analyzed along with a new CCV.
- **10.2.4** Procedure for Continuing Calibration:
  - **10.2.4.1** <u>**5ml Water:**</u> 12.5ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.
  - **10.2.4.2 <u>25ml Water:</u>** 5ul of target compound mixture is added to a 50ml volumetric flask. A 25ml aliquot is analyzed. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis.
  - **10.2.4.3** <u>Soil:</u> 25ul of target compound mixture is added to a 50ml volumetric flask. A 5ml aliquot is transferred to a sample vial. Internal standards and system monitoring compounds are added by the auto sampler prior to analysis

#### 10.3 <u>Sample Analysis</u>

- **10.3.1** BFB tuning criteria and GC/MS calibration verification must be met before sample analysis begins.
- **10.3.2** The acquisition time of the BFB tune establishes a 12hr. batch. The CCV, MSB, and VBLK must be analyzed within 12hrs, unless specified by the client request. The remaining time in the 12hr batch is utilized to run samples of similar matrix. The time of initiation of purging is considered the injection time. All aqueous samples are considered a water matrix. All solid samples, with the exception of sludges, are considered soil matrix. Sludges are run medium level.
- **10.3.3** Samples and standard solutions are brought to ambient temperature before analysis.
- **10.3.4** Prior to the analysis of samples, a method blank must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for method blanks must be met prior to sample analysis.

**10.3.5** Within the analytical batch an LCS must be analyzed in accordance with the associated procedures for a given matrix. Technical criteria for the LCS must be met with each batch.

#### 10.4 <u>Water Sample Analysis</u>

- **10.4.1** A 5ml sample aliquot is spiked with internal and system monitoring compounds to a final concentration of 25 ug/L each. 25ml analysis requires a final concentration of 10ug/L. The spike may be performed manually with a Hamilton gas tight syringe or the auto sampler may be used. The sample is then loaded onto the auto sampler where it is in turn transferred to the purge chamber.
- **10.4.2** The sample is purged for  $11.0 \pm 1$  minute at ambient temperature.
- **10.4.3** At the end of the purge time, the sample is desorbed onto the gas chromatograph column by rapidly heating the trap from 190C to 250<sup>o</sup>C (depending on manufacturer specifications) while the trap is back flushed with Helium between 20 60 ml/minute according to the manufactures specifications. The sample is desorbed onto the column and the gas chromatograph temperature ramping program is initiated.
- **10.4.4** While the trap is in the bake mode, the purge chamber is flushed with two 5ml aliquots of reagent water in order to avoid possible contamination from carryover of target compounds.
- **10.4.5** After the sample has desorbed, the trap is conditioned from 190°C to 260°C according to the manufactures specifications. After baking, the trap is ready for the next sample.
- **10.4.6** Dilutions may be necessary if the concentration of any target compound exceeds the working range of the calibration.
- **10.4.7** In the event that a dilution is required, a measured volume of sample is added to a volumetric flask then brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described.

#### 10.5 Low Level Soil/Sediment Sample Analysis

- **10.5.1** The low level soil method is based on a heated purge of a 5g sample mixed with reagent water containing a final concentration of 50 ug/L of internal and system monitoring compounds.
- **10.5.2** If a dilution of the soil/sediment is required, a smaller portion of soil may be used. The smallest amount of soil that may be used is 0.5g. If a higher dilution is required, the sample must be analyzed as a medium level soil/sediment.
- **10.5.3** Initial and continuing calibrations that are used for the quantitation of low soils/sediments are analyzed using the same purge and trap conditions as samples.
- **10.5.4** The sample consists of the entire contents of the sample container. A 5g portion is removed using a narrow metal spatula or wooden tongue depressor. This aliquot is then

placed into an empty VOA vial and the weight is recorded to the nearest 0.01g. 5ml of reagent water is added to the vial and it is capped.

- **10.5.5** Internal standards and system monitoring compounds are added to the sample immediately prior to heating and purging by the auto sampler.
- **10.5.6** After reagent water is added, the soil/sediment sample is heated to  $40^{\circ}C \pm 1^{\circ}C$  then purged for  $11 \pm 1$  minutes.
- **10.5.7** After purging, the sample is subjected to desorbing as described for water analysis.

#### 10.6 <u>Medium Level Soil/Sediment Samples</u>

- **10.6.1** The medium level soil/sediment method is based on an extraction of the sample with methanol. An aliquot of the extract is then added to a 50ml of reagent water.
- **10.6.2** The sample consists of the entire contents of the sample container. A 5g portion is removed using a narrow metal spatula or wooden tongue depressor. This aliquot is then placed into an empty 20ml vial and the weight is recorded to the nearest 0.01g.
- **10.6.3** 1ml of system monitor compound mixture is then added to the sample.
- **10.6.4** A 9ml aliquot of methanol is quickly added to the sample, bringing the final volume to 10ml. The vial is capped and the sample is shaken for 2 minutes.
- **10.6.5** A pre-determined amount of methanol extract is added to a 50ml volumetric flask, brought to volume with reagent water and inverted 3 times. The sample in the neck portion is discarded and the remainder of the sample is transferred into a 40ml VOA vial. Analysis may then proceed as previously described in section 10.4.
- **10.6.6** If sample extracts are prepared in the field (e.g. Terracore kits) then both system monitoring compounds and internal standards are added by the auto-sampler prior to analysis.
- **10.6.7** Table 3 may be used to determine the volume of methanol extract required for a given dilution factor.

#### 10.7 pH Determinations For Water Samples

**10.7.1** After the sample aliquots are taken from the VOA vials, the pH of the sample is determined using wide range pH paper. A checkmark will be entered in the injection logbook if the sample pH is <2, however if the sample demonstrates a pH>2, the actual pH will be noted in the injection logbook.

#### 11.0 <u>Calculations / Data Reduction</u>

#### 11.1 Calculations For MS/MSD Samples

11.1.1 The calculations to determine concentrations are the same equations described for

sample analysis of a given matrix.

**11.1.2** The percent recovery of the matrix spiking compounds is determined using equation:

Matrix Spike Recovery = 
$$\frac{SSR - SR}{SA}$$
 x 100

Where: SSR = Spiked sample result SR = Sample results SA = Spike added

**11.1.3** The relative percent difference (RPD) of the recoveries of each compound between the matrix spike and matrix spike duplicate is determined using equation:

 $RPD = \underbrace{|MSR - MSDR|}_{1/2} \times 100$ 

Where: MSR = Matrix spike recovery MSDR = Matrix spike duplicate recovery

#### 11.2 Calculations For Initial Calibration

**11.2.1** The relative response factor (RRF) for each target compound and each system monitoring compound is calculated using equation.

$$\begin{array}{c|c} \mathsf{RRF=} & \underline{\mathsf{Ax}} & \mathsf{x} & \underline{\mathsf{Cis}} \\ \hline \mathsf{Ais} & & \mathbf{Cx} \end{array}$$

Where,

- Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)
- Ais = Area of the characteristic ion (EICP for the specific internal standard (see Table 4) Cis = Concentration of the internal standard
- Cx = Concentration of the compound to be measured
- **11.2.2** The relative response factor of the Xylenes requires the use of the area response and the concentration of the peak that represents the single isomer.
- **11.2.3** The relative response factor of 1,2-dichloroethene is calculated using the sum of the areas of both isomers and the sum of the concentrations.
- **11.2.4** The average response factor (RRF) is calculated for all compounds of interest.
- **11.2.5** The relative standard deviation (% RSD) is calculated over the working range of the curve

for all compounds using equation:

 $\% \text{RSD} = \underline{\text{Standard Deviation}}_{\text{Mean}} \times 100$  MeanStandard Deviation =  $\sqrt{\frac{\sum_{i=1}^{n} (\chi i - \overline{\chi})2}{n-1}}$ 

Where,

Xi = each individual value used to calculate the mean

X = the mean of n values

n = the total number of values

## 11.3 <u>Calculations For Continuing Calibration</u>

- **11.3.1** The relative response factor (RRF) for all target compounds and system monitoring compounds is calculated using equation 11.2.1.
- **11.3.2** The percent difference between the initial calibration and the continuing calibration is determined for all target compounds and system monitoring compound using equation:

%Difference = <u>RRFc</u> - <u>RRFi</u> x 100 RRFi

Where,

RRFc = Relative response factor from continuing calibration standard RRFi = Mean relative response factor from the most recent initial calibration meeting technical acceptance criteria

## 11.4 Percent Moisture Determinations

**11.4.1** Immediately after weighing the sample for analysis, a 5-10g portion is weighed into a tarred aluminum weigh pan. The sample is then dried at 105<sup>o</sup>C. The sample is allowed to cool. The final weight is recorded. Using the equation for % moisture, concentrations relative to the dry weight of the soil/sediment samples, may be determined.

%moisture = <u>g of wet sample - g of dry sample</u> x 100 g of wet sample

- **11.5** <u>Quantitation of volatile target compounds</u> is done using the internal standard method. The Internal Standard RRF of the continuing calibration is used in the quantitation calculation.
- **11.5.1** <u>Water Samples:</u> The following equation is used to calculate water samples:

Concentration ug/L = (Ax) (Is) (DF)(Ais) (RRF) (Vo)

Where,

- Ax = Area of the characteristic ion (EICP) for the compound to be measured (see Table 4)
- Ais = Area of the characteristic ion (EICP) for the specific internal standard (see Table 4) Is = Amount of internal standard added in nanograms (ng)
- RRF= Relative response factor from the ambient temperature purge of the calibration standard.
- Vo = Volume of water purged in milliliters (mL)
- Df = Dilution factor. The dilution factor for analysis of water samples for volatiles by this method is defined as the ratio of the number of milliliters (mL) of water purged (i.e., Vo above) to the number of mL of the original water sample used for purging. For example, if 2.0 mL of sample is diluted to 5 mL with reagent water and purged, Df = 5 mL/2.0 mL = 2.5. If no dilution is performed, Df = 1.
- **11.5.2** <u>Low Level Soil/Sediment Samples</u> The following equation is used for low level soil/sediment samples:

Concentration ug/Kg (dry weight basis) = (Ax) (Is)(Ais) (RRF) (Ws) (D)

Where,

Ax, Is, Ais are as given for water. RRF = Relative response factor form the heated purge of the calibration standard. D =  $\frac{100 - \% \text{ moisture}}{100}$ Ws = Weight of sample added to the purge tube, in grams (g).

## 11.5.3 Medium Level Soil/Sediment Samples

The following equation is used for quantitation of medium level soil/sediment samples:

Concentration ug/Kg (Dry weight basis) = (Ax) (Is) (Vt) (1000) (Df)(Ais) (RRF) (Va) (Ws) (D)

Where,

Ax, Is, Ais are as given for water.

- RRF = Relative response factor from the ambient temperature purge of the calibration standard.
- Vt = Total volume of the methanol extract in milliliters (mL).

NOTE: This volume is typically 10 mL, even though only 1 mL is transferred to the vial.

- Va = Volume of the aliquot of the sample methanol extract (i.e., sample extract not including the methanol added to equal 100 uL) in micro liters (ul) added to reagent water for purging.
- Ws = Weight of soil/sediment extracted, in grams (g).

 $D = \frac{100 - \% \text{ moisture}}{100}$ 

Df = Dilution factor. The dilution factor for analysis of soil/sediment samples for volatiles by the medium level method is defined as:

ul most conc. extract used to make dilution + ul clean solvent ul most conc. extract used to make dilution

(The dilution factor is equal to 1.0 in all cases other than those requiring dilution of the sample methanol extract (Vt). The factor of 1,000 in the numerator converts the value of Vt from mL to ul.)

- **11.6** When quantitating the sample concentration of Xylenes (total), the areas of both the m & p Xylene peak and the o-Xylene peak are summed and the RRF determined using equation 11.2.1 are used. The concentration of each peak may be determined separately and then summed to determine the concentration of Xylene (total).
- **11.7** When quantitating the concentration of 1,2-Dichloroethene (total), the concentrations of the two isomers (cis and trans) are summed.
- **11.8** Secondary ion quantitation may be used if interferences (such as matrix effects) may cause a bias in quantitation.
- **11.9** If manual integration of any compound (including internal standards, system monitoring compounds, target or tentatively identified compounds) is required, the EICP of that compound will be provided. All manual integrations will be identified with an "m" and initialed and dated by the GC/MS analyst.

#### 11.10 Tentatively Identified Compounds

- **11.10.1** An estimated concentration for tentatively identified compounds will be determined using the equations described above for a given matrix using the total area counts of both the tentatively identified compound and the nearest internal standard which is free of interferences.
- **11.10.2** The RRF used to determine all concentrations of tentatively identified compounds will be an assumed RRF of one (1).
- **11.10.3** All tentatively identified compounds will be qualified as "J" (estimated) and "N" (presumptive evidence).

#### 11.11 System Monitoring Compounds

**11.11.1** The recovery of all system monitoring compounds in samples, blanks matrix spikes and matrix spike duplicates, is calculated using equation:

#### % Recovery = <u>Concentration (amount) found</u> x 100 Concentration (amount) spiked

- **11.11.2** The recovery limits for each system monitoring compound are laboratory established on an annual basis. The recoveries must be within the criteria limits. If they fall outside criteria limits, the results must be evaluated and the sample reanalyzed, if necessary.
- **11.11.3** The relative retention time (RRT) of each system monitoring compound must be within the acceptance windows of ±0.06 RRT.

#### 11.12 Internal Standards

- **11.12.1** The internal standards of all samples, blanks, matrix spikes and matrix spike duplicates must be monitored. The EICP area of each internal standard must be within the range of -50.0 percent to 200.0 percent of those in the continuing calibration.
- **11.12.2** The relative retention time (RRT) of each internal standard must be within 0.5 minutes (30 seconds) of those in the continuing calibration.

#### 11.13 Verification of Calculated Result

**11.13.1** The laboratory analyst/data entry analyst will print out and review sample worksheets and hand calculate the result for positive hits, internal standards and surrogates for comparison to the LIMS calculated result. Corrective action will result, if needed.

#### 12.0 <u>Method Performance</u>

Each analyst prior to sample analysis will perform 4 replicate QC check standards as an Initial Demonstration of Capability. The average recovery and standard deviation are calculated in the LIMS system and kept with each analyst's training file.

#### 12.1 <u>Method Detection Limit Study (MDL)</u>

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in the QA Manual. MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

## 12.2 Demonstration of Capabilities

- **12.2.1** A one-time initial demonstration of performance for each individual method for both soils and water matrices must be generated.
- **12.2.2** This requires quadruplicate analysis of a mid–level check standard containing all of the standard analytes for the method using the same procedures used to analyze samples, including sample preparation.
- **12.2.3** Compare these results with the acceptance criteria given in the Method or to laboratory historical limits (if available).
- **12.2.4** Repeat the test for any analyte that does not meet the acceptance criteria. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

#### 12.3 <u>Training Requirements</u>

- **12.3.1** The supervisor has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.
- **12.3.2** The following analyst validation information is maintained for this method in the laboratory QA files.
- **12.3.3** The analyst must complete the laboratory safety orientation training that includes, but is not limited to, chemicals, PPE requirements, and electrical safety.
- **12.3.4** The analyst must read and understand this SOP.
- **12.3.5** The analyst must read and understand the Method used as reference for this SOP.
- **12.3.6** The analyst must complete a DOC or successfully analyze PT samples annually.
- **12.3.7** The analyst must complete the TestAmerica Quality Assurance Training.

#### 13.0 Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

#### 14.0 <u>Waste Management</u>

Waste management practices are conducted consistent with all applicable rules and regulations.

Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to the Corporate Safety Manual. The following waste streams are produced when this method is carried out.

## 14.1 <u>Waste Streams Produced by the Method</u>

The following waste streams are produced when this method is carried out.

- **14.1.1** <u>Spill Response:</u> Any spills must be cleaned up immediately and handled correctly. Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.
- 14.1.2 <u>Aqueous waste generated from analysis:</u> Any wastes that have a pH < 7 must be disposed of in an "A" waste container. Any wastes having a pH > 7 must be disposed of in a "D" waste container.
- **14.1.3** <u>Solvent waste generated from analysis:</u> Solvent waste is stored in laboratory approved metal waste receptacle and labeled "C" waste. Waste receptacles are then taken to sample control where they are then properly disposed of.
- **14.1.4** <u>Solid waste generated from analysis:</u> Solid volatile analysis waste consists of soils and glass. The soil is wrapped in tin foil and placed in the solid waste receptacle. Soils used for dry weight measurements are also disposed of in this manner. Glass waste such as pipettes and vials are rinsed and disposed of in approved glass receptacles
- **14.1.5** <u>Expired Standards:</u> Expired and used standards are stored in a laboratory approved metal waste receptacle labeled "BV". Waste receptacles are then taken to sample control where they are then properly disposed of.

#### 15.0 <u>References / Cross-References</u>

• Method 8260B, "Test Methods for Evaluating Solid Waste"; SW846, Third Edition, December 1996.

#### 16.0 <u>Method Modifications:</u>

ltem	Method	Modification
		N/A

#### 17.0 <u>Attachments</u>

Table 1. Compounds Determined by Method 8260B

Table 2. BFB Key lons and Ion Abundance Criteria

Table 3. Volume of Medium Level Extracts for Dilution

Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds

 Table 5. Poor Performing Compounds

 Table 6. Job Summary Check List (Page 1 & 2)

Tables 7-15. Composition of Stock Standards

#### 18.0 <u>Revision History</u>

- Revision 0, dated 18 July, 2008
  - Quality Director change, signature added
  - Section 6.0: Changed section to reflect current instrumentation and column specifications
  - Section 9: Added Trichloroethene to MSB list, took out duplicate Chlorobenzene
  - Minor grammatical changes
- Revision 01, dated -24 July, 2008
  - Sec 2.4- removed reference to jet separator and added clarification to 5 point curve reference
  - Section 7.5.5.2.1: Changed volumes used to make ICAL
  - Reduced throughout document volumes used from 25ul to 12.5ul. Also changed throughout final concentrations from 50ppb to 25ppb.
  - Sec 9.2.3 and 9.5.5.1- replaced couple with few
  - Sec 9.4.1.2- removed reference to use of mean RSD
  - Sec 9.5.1.1- added text to clarify lab practice
  - Sec 10.1.3- fixed table to reflect concentrations and volumes used.
  - Sec 10.4.3- added temperature range due to variances in instrumentation
- Revision 02, dated 30 March, 2010
  - SOP revised. All sections.
  - Added table 5 for poor performing compounds and their criteria section 9.2.4.

	Appropriate Technique						
Compound	CAS No. <sup>b</sup>	5030/5035	5031	5032	5021	5041	Direct Injection
Acetone	67-64-1	рр	С	С	nd	с	с
Acetonitrile	75-05-8	рр	С	nd	nd	nd	С
Acrolein	107-02-8	рр	С	С	nd	nd	С
Acrylonitrile	107-13-1	рр	С	С	nd	С	С
Allyl alcohol	107-18-6	ht	С	nd	nd	nd	С
Allyl chloride	107-05-1	С	nd	nd	nd	nd	С
Benzene	71-43-2	С	nd	С	С	С	С
Benzyl chloride	100-44-7	С	nd	nd	nd	nd	С
Bis(2-chloroethyl)sulfide	505-60-2	рр	nd	nd	nd	nd	С
Bromoacetone	598-31-2	рр	nd	nd	nd	nd	С
Bromochloromethane	74-97-5	С	nd	с	с	с	с
Bromodichloromethane	75-27-4	С	nd	с	с	с	с
4-Bromofluorobenzene (surr)	460-00-4	с	nd	С	С	С	С
Bromoform	75-25-2	С	nd	С	С	С	С
Bromomethane	74-83-9	С	nd	С	с	с	С
n-Butanol	71-36-3	ht	С	nd	nd	nd	С
2-Butanone (MEK)	78-93-3	рр	С	с	nd	nd	с
t-Butyl alcohol	75-65-0	рр	С	nd	nd	nd	с
Carbon disulfide	75-15-0	рр	nd	С	nd	С	с
Carbon tetrachloride	56-23-5	С	nd	С	с	с	с
Chloral hydrate	302-17-0	рр	nd	nd	nd	nd	с
Chlorobenzene	108-90-7	С	nd	С	С	С	С
Chlorobenzene-d5 (IS)		С	nd	с	с	с	с
Chlorodibromomethane	124-48-1	С	nd	С	nd	С	С
Chloroethane	75-00-3	С	nd	С	с	с	С
2-Chloroethanol	107-03-3	рр	nd	nd	nd	nd	С
2-Chloroethyl vinyl ether	110-75-8	С	nd	С	nd	nd	С
Chloroform	67-66-3	С	nd	С	с	С	С
Chloromethane	74-87-3	С	nd	С	С	С	С
Chloroprene	126-99-8	С	nd	nd	nd	nd	С
3-Chloropropionitrile	542-76-7	I	nd	nd	nd	nd	рс
Crotonaldehyde	4170-30-3	рр	С	nd	nd	nd	С
1,2-Dibromo-3- chloropropane	96-12-8	рр	nd	nd	С	nd	с
1,2-Dibromoethane	106-93-4	с	nd	nd	с	nd	с
Dibromomethane	74-95-3	С	nd	С	С	с	с
1,2-Dichlorobenzene	95-50-1	с	nd	nd	с	nd	С

# Table 1: Compounds Determined by Method 8260B

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		Appropriate Technique					
Compound	CAS No. <sup>b</sup>	5030/5035	5031	5032	5021	5041	Direct Injection
1,3-Dichlorobenzene	541-73-1	С	nd	nd	С	nd	С
1,4-Dichlorobenzene	106-46-7	С	nd	nd	С	nd	С
1,4-Dichlorobenzene-d4 (IS)		С	nd	nd	С	nd	С
cis-1,4-Dichloro-2-butene	1476-11-5	С	nd	С	nd	nd	С
trans-1,4-Dichloro-2- butene	110-57-6	рр	nd	С	nd	nd	С
Dichlorodifluoromethane	75-71-8	С	nd	С	С	nd	С
1,1-Dichloroethane	75-34-3	С	nd	С	С	С	С
1,2-Dichloroethane	107-06-2	С	nd	С	С	С	С
1,2-Dichloroethane-d4 (surr)		С	nd	С	С	С	С
1,1-Dichloroethene	75-35-4	С	nd	С	С	С	С
trans-1,2-Dichloroethene	156-60-5	С	nd	С	с	С	С
1,2-Dichloropropane	78-87-5	С	nd	С	с	С	С
1,3-Dichloro-2-propanol	96-23-1	рр	nd	nd	nd	nd	С
cis-1,3-Dichloropropene	10061-01-5	С	nd	С	nd	С	С
trans-1,3-Dichloropropene	10061-02-6	С	nd	С	nd	С	С
1,2,3,4-Diepoxybutane	1464-53-5	С	nd	nd	nd	nd	С
Diethyl ether	60-29-7	С	nd	nd	nd	nd	С
1,4-Difluorobenzene (I.S.)	540-36-3	nd	nd	nd	nd	С	С
1,4-Dioxane	123-91-1	рр	С	С	nd	nd	С
Epichlorohydrin	106-89-8	I	nd	nd	nd	nd	С
Ethanol	64-17-5	I	С	С	nd	nd	С
Ethyl acetate	141-78-6	I	С	nd	nd	nd	С
Ethylbenzene	100-41-4	С	nd	С	с	С	С
Ethylene oxide	75-21-8	рр	С	nd	nd	nd	С
Ethyl methacrylate	97-63-2	С	nd	С	nd	nd	С
Fluorobenzene (IS)	462-06-6	С	nd	nd	nd	nd	nd
Hexachlorobutadiene	87-68-3	С	nd	nd	с	nd	с
Hexachloroethane	67-72-1	I	nd	nd	nd	nd	С
2-Hexanone	591-78-6	рр	nd	С	nd	nd	С
2-Hydroxypropionitrile	78-97-7	I	nd	nd	nd	nd	рс
lodomethane	74-88-4	С	nd	С	nd	С	с
Isobutyl alcohol	78-83-1	рр	С	nd	nd	nd	с
Isopropylbenzene	98-82-8	С	nd	nd	с	nd	с
Malononitrile	109-77-3	рр	nd	nd	nd	nd	с
Methacrylonitrile	126-98-7	рр	I	nd	nd	nd	с
Methanol	67-56-1	I	С	nd	nd	nd	с
Methylene chloride	75-09-2	С	nd	С	С	С	С

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	Appropriate Technique						
Compound	CAS No. <sup>b</sup>	5030/5035	5031	5032	5021	5041	Direct Injection
Methyl methacrylate	80-62-6	С	nd	nd	nd	nd	С
4-Methyl-2-pentanone (MIBK)	108-10-1	рр	С	С	nd	nd	С
Naphthalene	91-20-3	С	nd	nd	С	nd	С
Nitrobenzene	98-95-3	С	nd	nd	nd	nd	С
2-Nitropropane	79-46-9	С	nd	nd	nd	nd	С
N-Nitroso-di-n-butylamine	924-16-3	рр	С	nd	nd	nd	С
Paraldehyde	123-63-7	рр	С	nd	nd	nd	С
Pentachloroethane	76-01-7	I	nd	nd	nd	nd	С
2-Pentanone	107-87-9	рр	С	nd	nd	nd	С
2-Picoline	109-06-8	рр	С	nd	nd	nd	С
1-Propanol	71-23-8	рр	С	nd	nd	nd	С
2-Propanol	67-63-0	рр	С	nd	nd	nd	С
Propargyl alcohol	107-19-7	рр		nd	nd	nd	С
B-Propiolactone	57-57-8	рр	nd	nd	nd	nd	С
Propionitrile (ethyl cyanide)	107-12-0	ht	С	nd	nd	nd	С
n-Propylamine	107-10-8	С	nd	nd	nd	nd	С
Pyridine	110-86-1	I	С	nd	nd	nd	С
Styrene	100-42-5	С	nd	С	С	С	С
1,1,1,2-Tetrachloroethane	630-20-6	С	nd	nd	С	С	С
1,1,2,2-Tetrachloroethane	79-34-5	С	nd	С	С	С	С
Tetrachloroethene	127-18-4	С	nd	С	С	С	С
Toluene	108-88-33	С	nd	С	С	С	С
Toluene-d8 (surr)	2037-26-5	С	nd	С	С	С	С
o-Toluene	95-53-4	рр	С	nd	nd	nd	С
1,2,4-Trichlorobenzene	120-82-1	С	nd	nd	С	nd	С
1,1,1-Trichloroethane	71-55-6	С	nd	С	С	С	С
1,1,2-Trichloroethane	79-00-5	С	nd	С	С	С	С
Trichloroethane	79-01-6	С	nd	С	С	С	С
Trichlorofluoromethane	75-69-4	С	nd	С	С	С	С
1,2,3-Trichloropropane	96-18-4	С	nd	С	С	С	С
Vinyl acetate	108-05-4	С	nd	С	nd	nd	С
Vinyl chloride	75-01-4	С	nd	С	С	С	С
Xylene (Total)	1330-20-7	С	nd	С	С	С	С

c= b= Adequate response by this technique

Chemical Abstract Services Registry Number Poor purging efficiency resulting in high EQLs pp= I=

Inappropriate technique for this analyte

Not determined nd=

Surrogate surr=

IS= Internal Standard

ht= Method analyte only when purged at 80 C

#### pc= Poor chromatographic behavior

The following compounds are also amenable to analysis by Method 8260:

## Table 2. BFB Key lons and lon Abundance Criteria

Required Intensity (relative abundance)	
15 to 40% of m/z 95	
30 to 60% of m/z 95	
Base peak, 100% relative abundance	
5 to 9% of m/z 95	
less than 2% of m/z 174	
Greater than 50% of m/z 95	
5 to 9% of m/z 174	
Greater than 95% but less than 101% of m/z 174	
5 to9% of m/z 176	
	15 to 40% of m/z 95 30 to 60% of m/z 95 Base peak, 100% relative abundance 5 to 9% of m/z 95 less than 2% of m/z 174 Greater than 50% of m/z 95 5 to 9% of m/z 174 Greater than 95% but less than 101% of m/z 174

\*Alternate tuning criteria may be used, (e.g. CLP, Method 524.2, or manufacturers' instructions), provided that method performance is not adversely affected.

#### Table 3. Volume of Medium Level Extracts for Dilution

Dilution Factor	Volume of Extract
1	100ul
2	50ul
5	20ul
10	10ul
20	5ul
25	4ul
40	2.5ul
50	2ul
100	1ul
200	50ul of a 1/10 Dilution

Primary Characteristic Ion	Secondary Characteristic Ion(s
58	43
41	40,39
56	55,58
53	52,51
57	58,39
76	41,39,78
78	-
91	126,65,128
136	43,138,93,95
156	77,158
128	49,130
83	85,127
173	175,254
94	96
74	43
56	41
72	43
91	92,134
105	134
119	91,134
76	78
117	119
82	44,84,86,111
48	75
112	77,114
56	49
129	208,206
64 (49*)	66 (51*)
49	44,43,51,80
	111,158,160
	65,106
	85
	52 (51*)
	88,90,51
	49,89,91
	126
	126
75	155,157
129	127
107	109,188
93	95,174
146	111,148
	115,150
	111,148
	111,148
75	53,77,124,89

# Table 4. Characteristic Masses (m/z) for Purgeable Organic Compounds

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Dichlorodifluoromethane	85	87
1,1-Dichlorothane	63	65,83
1,2-Dichloroethane	62	98
1,1-Dichlorothene	96	61,63
cis-1,2-Dichloroethene	96	61,98
trans-1,2-Dichloroethene	96	61,98
1,2-Dichloropropane	63	112
1,3-Dichloropropane	76	78
2,2-Dichloropropane	77	97
1,3-Dichloro-2-propanol	79	43,81,49
1,1-Dichloropropene	75	110,77
cis-1,3-Dichloropropene	75	77,39
trans-1,3-Dichloropropene	75	77,39
1,2,3,4-Diepoxybutane	55	57,56
Diethyl ether	74	45,59
1,4-Dioxane	88	
*		58,43,57
Epichlorohydrin	57	49,62,51
Ethanol	31	45,27,46
Ethyl acetate	88	43,45,61
Ethylbenzene	91	106
Ethylene oxide	44	43,42
Ethyl methacrylate	69	41,99,86,114
Hexachlorobutadiene	225	223,227
Hexachloroethane	201	166,199,203
2-Hexanone	43	58,57,100
2-Hydroxypropionitrile	44	43,42,53
lodomethane	142	127,141
Isobutyl alcohol	43	41,42,74
Isopropylbenzene	105	120
p-Isopropyl toluene	119	134,91
Malonitrile	66	39,65,38
Methacrylonitrile	41	67,39,52,66
Methyl acrylate	55	85
Methyl-t-butyl ether	73	57
Methylene chloride	84	86,49
Methyl ethyl ketone	72	43
Methyl iodide	142	127,141
Methyl methacrylate	69	41,100,39
4-Methyl-2-pentanone	100	43,58,85
Naphthalene	128	
Nitrobenzene	123	51,77
	46	51,77
2-Nitropropane 2-Picoline	93	- 66,92,78
Pentachloroethane	167	130,132,165,169
Propargyl alcohol	55	39,38,53
B-Propiolactone	42	43,44
Propionitrile (ethyl cyanide)	54	52,55,40
n-Propylamine	59 91	41,39 120

Analyte	Primary Characteristic Ion	Secondary Characteristic Ion(s)
Pyridine	79	52
Styrene	104	78
1,2,3-Trichlorobenzene	180	182,145
1,2,4-Trichlorobenzene	180	182,145
1,1,1,2-Tetrachloroethane	131	133,119
1,1,2,2-Tetrachloroethane	83	131,85
Tetrachloroethene	164	129,131,166
Toluene	92	91
1,1,1-Trichloroethane	97	99,61
1,1,2-Trichloroethane	83	97,85
Trichloroethene	95	97,130,132
Trichlorofluoromethane	151	101,153
1,2,3-Trichloropropane	75	77
1,2,4-Trimethylbenzene	105	120
1,3,5-Trimethylbenzene	105	120
Vinyl acetate	43	86
Vinyl chloride	62	64
o-Xylene	106	91
m-Xylene	106	91
p-Xylene	106	91
I	NTERNAL STANDARDS/SURRO	GATES
Benzene-d6	84	83
Bromobenzene-d5	82	162
Bromochloromethane-d2	51	131
1,4-Difluorobenzene	114	
Chlorobenzene-d5	117	
1,4-Dichlorobenzene-d4	152	115,150
1,1,2-Trichloroethane-d3	100	
4-Bromofluorobenzene	95	174,176
Chloroform-d1	84	, , , , , , , , , , , , , , , , , , ,
Dibromofluoromethane	113	

## Table 5. Poor Performing Compounds

1,1-Dimethoxyethane*	Bromomethane
1,2-Dibromo-3-chloropropane (DBCP)	Carbon Disulfide
1,4-Dioxane*	Chloroethane
2-Butanone (MEK)	Cyclohexanone*
2-Chloroethylvinyl ether	Dichlorodifluoromethane
2-Nitropropane*	lodomethane
4-Methyl-2-pentanone (MIBK)	Methyl Acetate
Acetone	Propylene Oxide*
Acrolein	trans-1,4-Dichloro-2-butene

\* Indicates "Add" compounds that are not routinely spiked for in LCS/MS/SD

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		a.a. (1997)					
	GCMS Voa	Work Order Summary					
Work Order:	_			Method:			
Work Order Due:							
Sequence #1							
Batch ID	Sequence ID	• n.	Instrument		Date Created		
Sequence #2							
Batch ID	Sequence ID		Instrument		Date Created		
Sequence #3		a da se se se					
Batch ID	Sequence ID		Instrument		Date Created		
Sequence #4							
Batch ID	Sequence ID		Instrument		Date Created		
Sequence #5							
Batch ID	Sequence ID		Instrument		Date Created		
Sequence #6							
Batch ID	Sequence ID		Instrument		Date Created		
Analyte Comments:					and the second state of th		
Sample Comments:							
Data Tooled And Analyst Rev	iewed:	Initials		Date			
-	1011041						
Second Review:		Initials:		Date:			
Check Second Review							

## Table 6. Job Summary Check List

Check Second Review:

Quantitative Accuracy

\_\_\_\_ QC Samples

Method and/or Quapp Specific QC Criteria Manual Integrations Reviewed

GCMS DATA Analysis Checklist Revision 0

02/20/2009

Qualifier ID	Qualifiers
D03	Dilution for foaming
D04	Dilution for nontargets (TICs)
D05	Diluted for matrix effect on IS
D06	Diluted for matrix effect on Surr
D07	Diluted for TCLP matrix
D08	Diluted for targets
D11	Diluted for insuficient Volume (Needs Job Exception)
D13	Diluted for other reason
P-HS	Sample contained headspace. (Needs Job Exception)
1	IS out. Re-analysis confirms matrix.
Z	Surr out due to Matrix
P-6	PH greater than 2 analyzed within seven days
S-10	Insuficient volume for reshot (Needs Job Exception)
B-2	VBLK contaminated with Nontarget analyte
B-1	VBlk contaminated with target analyte, at a level less than 10X sample detection
B-3	VBlk contaminated with target analyte, at a level at a level above reporting limit
L	MSB was out high samples ND
L-1	MSB was out high.
L-2	MSB was out low.
L-4	MSB out low results Blased low
NI	See case narrative
P-11	PH greater than 2 (Needs Job Exception)
H	Analyzed past HT. (Needs Job Exception)
M7	MS/SD Above acceptance limits. See LCS
M8	MS/SD Below acceptance limits. See LCS
R9	Sample RPD exceeded limits (Use for MS/SD that exceedes RPD)

#### Additional Comments

## Table 7. Gas Mixture

				رىقىچى يەر
Certifi	cate o	f Compo	osition	
DESCRIPTION: Volatile Organic Compo	-	بی <i>لد '</i>		
CATALOG NO.: 48799-U		MFG DATE: N	10v-2005	. 72 8-20 . 73 1-7
LOT NO.: LB34727	1	EXPIRATION DATE: F	12007 MVSC.	- 73 1-+
SOLVENT: METHANOL				
ANALYTE (1)	CAS	PERCENT PURITY (2)	WEIGHT CONCENTRATION	SUPELCO (3) LOT NO
BROMOMETHANE	74-83-9	99.9 (a)	2000	LB22203
HLOROETHANE	75-00-3	98.7 (a)	2000	LB29285
CHLOROMETHANE	74-87-3	99.9 (a)	2000	LA66620
DICHLORODIFLUOROMETHANE RICHLOROFLUOROMETHANE	75-71-8	99.9 (a)	2000	LB24923
INTL CHLOROFLUOROMETHANE	75-69-4	99.9 (a)	2000	LA79530
STEPALDA	75-01-4	99.9	2000	LB18727
<ol> <li>Listed in alphabetical order.</li> <li>Determined by capillary GC-FID, un]         <ul> <li>a) GC; detector HALL</li> <li>NIST traceable weights are used to Concentration of analyte in solutic Class A volumetric glassware. Weig</li> </ul> </li> </ol>	verify balanc on is ug/ml +/	e calibration wit ~ 0.5%, uncertain	ty based upon bala	ince and
Flwood Doughty JA Manager upeloo warrants that its products conform to the inform urchaser must determine the suitability of the product for its talog or order invoice and packing slip for additional term	s particular use. P	lease see the latest	595 Nort	SUPELCO

DESCRIPTION:         502/524 Volatile Organics Calibration Mix           CATALOS NO.:         502111         NTS DATS:         KOV-2003           LOT NO.:         BL6275         EXTENSION DATE:         MARTINE           SOUTHT:         KETANON         KETANON         MARTINE         MARTINE           MINITY         (1)         KUMER         FULLATION DATE:         MARTINE           MINITY         (1)         KUMER         FULLATION DATE:         MARTINE           MINITY         (1)         KUMER         FULLATION         MARTINE           MINITY         (1)         KUMER         FULLATION         FULLATION         FULLATION           MINITY         (1)         KUMER         FULLATION         FULATION         FULLATION         FULLATION	15-0
HY.Y.       H.S.Y.       EXPERIENCE MARKET         HY.Y.       H.S.Y.       H.S.Y.       EXPERIENCE MARKET         MARKET       M.S.Y.       H.S.Y.       H.S.Y.       M.S.Y.         MARKET       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.         MARKET       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.         MARKET       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.       M.S.Y.         MARKET       M.S.Y.       M.S.Y.Y.       M.S.Y.       M.S.Y.       M.S	12 2 01
SUPER: NETHONS         NAME       1	
ALTER         ALTE         ALTE <t< td=""><td></td></t<>	
NUMER     FULLY (1)     NUMER     FULLY (2)     COMENTATION     Data       ENERGY     104-61     99.9     2000     2009     +/-     13       ENERGY     106-61     99.9     2000     2009     +/-     13       ENERGY     106-61     99.9     2000     1067     +/-     13       ENERGY     106-101-5     90.1     2000     2001     +/-     14       CHLOROENTENNE     106-101-5     90.1     2001     2022     +/-     11       DIERGYCHLOROENTENNE     106-101-4     90.5     2000     2001     +/-     16       DIERGYCHLOROENTENNE     106-101-4     90.5     2000     2014     +/-     13       DIERGYCHLOROENTENNE     106-61-3     90.8     2000     2014     +/-     14       DIERGYCHLOROENTENNE     106-61-3     90.8     2000     2014     +/-     15       DIERGYCHLOROENTENNE     106-61-3     90.9     2001     2022 <td< td=""><td></td></td<>	
EROMOGENZENE         109-86-1         99.9         2000         2009         +/-         17           BROMOCHLOROMETTANE         74-97-5         99.7         2000         1967         +/-         33           BROMOCHLOROMETTANE         75-27-4         99.9         2000         1967         +/-         38           CHEDNOFORM         75-27-4         99.9         2000         1960         +/-         38           CHEDNOFORM         75-27-4         99.9         2001         2008         +/-         18           CHEDNOFORM         67-66-3         99.9         2001         2021         +/-         18           CHEJ, 2-DICHLOROFENTENS (1)         10061-01-5         96.1         2000         1947         +/-         46           DIRROMOCHLOROMETTANE         124-48-1         99.9         2001         2022         +/-         18           DIRROMOCHLOROMETTANE         106-41-4         99.5         2000         2021         +/-         18           DIRROMOCHLOROMETTANE         106-41-4         99.5         2000         2021         +/-         18           DIRROMOCHLOROMETTANE         106-42-3         99.9         2001         2028         +/-         19	SUPE LOT
EROMOGENZENE         109-86-1         99.9         2000         2009         +/-         17           BROMOCHLOROMETTANE         74-97-5         99.7         2000         1967         +/-         33           BROMOCHLOROMETTANE         75-27-4         99.9         2000         1967         +/-         38           CHEDNOFORM         75-27-4         99.9         2000         1960         +/-         38           CHEDNOFORM         75-27-4         99.9         2001         2008         +/-         18           CHEDNOFORM         67-66-3         99.9         2001         2021         +/-         18           CHEJ, 2-DICHLOROFENTENS (1)         10061-01-5         96.1         2000         1947         +/-         46           DIRROMOCHLOROMETTANE         124-48-1         99.9         2001         2022         +/-         18           DIRROMOCHLOROMETTANE         106-41-4         99.5         2000         2021         +/-         18           DIRROMOCHLOROMETTANE         106-41-4         99.5         2000         2021         +/-         18           DIRROMOCHLOROMETTANE         106-42-3         99.9         2001         2028         +/-         19	1 LB03
BROMOCHLOROMETHANE       74-97-5       99.7       2000       1967       +/-       33         BROMOCHLOROMETHANE       75-27-4       99.9       2000       1744       +/-       38         CAREON TETRACHLORIDE       56-23-5       99.9       2000       1960       +/-       13         CHLOROMENNZENE       108-90-7       99.9       2001       2029       +/-       14         CHLOROMENNZENE       108-90-7       99.9       2000       2000       +/-       14         CHLOROMENNZENE       106-10-15       96.1       2000       2022       +/-       14         CHLOROMENLONG       1144-48-1       99.9       2011       2022       +/-       12         DIREMOMENTIANE       124-48-1       99.8       2000       2000       +/-       38         HEXACHLOROMENTIANE       104-61-4       99.5       2000       2014       +/-       45         IDGEOROMENTIANE       104-61-4       99.7       2000       2014       +/-       14         IDREMOMENTIANE       104-61-8       99.7       2000       2012       +/-       14         IDREMOMENTIANE       106-21-3       99.8       2001       2024       +/-	4 LA97
BROMOFORM         75-25-2         99.9         2000         1974         +/-         18           CAREON TETRACHLORIDE         56-23-5         99.9         2001         2020         +/-         14           CHLOROFORM         67-66-3         99.9         2000         2000         +/-         18           CIS 1, 3-DICHLOROFROPENE (2)         10061-01-5         96.1         2000         2022         +/-         18           CIS 1, 2-DICHLOROFROPENE (2)         10061-01-5         96.1         2000         2001         +/-         22           CIS 1, 2-DICHLOROFROPENE (2)         1068-00-1         90.8         2000         2000         +/-         38           DIEROMOCHLOROMETHANE         124-48-1         99.9         2001         2022         +/-         11           DIEROMORTIDIENE         74-95-3         98.8         2000         2014         +/-         28           HEXACHLOROBOTADIENE         100-41-4         99.5         2000         1957         +/-         28           ISOPROPYLIBENZENE         104-51-8         98.7         2000         1957         +/-         28           NEMETHYLENE         133-65-1         99.9         2001         2022         +/- <td>3 LA67</td>	3 LA67
BRONGFORM         75-25-2         99.9         2000         1974         +/-         88           CARRON TETRACHLORIDE         56-23-5         99.9         2001         2020         +/-         14           CHLOROFORM         67-66-3         99.9         2000         2000         +/-         18           CIS.1.3-DICHLOROFORMER (Z)         10061-01-5         96.1         2000         2022         +/-         18           CIS.1.2-DICHLOROFORMER (Z)         10061-01-5         96.1         2000         2022         +/-         11           DIBROMOCHLOROMETHAINE         126-59-2         97.6         2000         2000         +/-         43           ETHYLIBENZENE         100-41-4         99.5         2000         2000         +/-         45           ISOPROFYLIBENZENE (CUMENE)         98-82-8         99.0         2000         2012         +/-         17           MEXIMURATIONE (5)         108-36-1         99.9         2001         1956         +/-         28           N-PHOTHENNERENE         101-61-6         99.9         2001         2023         +/-         20           N-STYLENNE (5)         106-642-3         99.9         2000         1956         +/-	1 LB15
CHLOROPENEENE       108-90-7       99.9       2001       2029       +/-       14.         CHLOROPFORM       67-66-3       99.9       2000       2000       +/-       14.         CHS 1, 3-DICHLOROPTENE (Z)       10061-01-5       96.1       2000       2024       +/-       14.         CHS 1, 2-DICHLOROPTIVIENE       156-59-2       97.6       2000       1947       +/-       26.         DIBROMCHLOROSTIVIENE       156-59-2       97.6       2000       2000       +/-       13.         DIBROMCHLOROSTIVIENE       164-48-1       99.5       2000       2000       +/-       13.         STIVILEEXENE       100-41-4       49.5       2000       2000       +/-       45.         ISOROFYLBENZENE       104-51-8       98.2       2001       1946       +/-       45.         ISOROFYLBENZENE       104-51-8       99.0       2000       1957       +/-       28.         N-BUTILEENZENE       104-51-8       90.7       2001       2026       +/-       15.         N-PEOFYLEENZENE       103-65-1       99.9       2000       1950       +/-       39.         N-PEOFYLEENZENE       103-65-4       99.9       2000       1966 </td <td>7 LB15</td>	7 LB15
CHLOROPOPM       67-66-3       99.9       2000       2000       +/-       19.         CIS 1, 3-DICHLOROPOPME (2)       10061-01-5       96.1       2000       1947       +/-       26.         CIS-1, 2-DICHLOROPOTHYLENE       126-69-2       97.6       2000       1947       +/-       26.         DIERCMOCHLOROMETHANE       124-48-1       99.9       2001       2022       +/-       11.         DIERCMOCHLOROMETHANE       74-95-3       99.8       2000       2000       +/-       48.         ENDROMENTANE       106-41-4       99.5       2000       2014       +/-       45.         ISOPROFYLMENEE       106-41-4       99.5       2000       2012       +/-       15.         ISOPROFYLMENEENE       106-41-4       99.5       2000       1956       +/-       45.         ISOPROFYLMENEENE       106-41-51-8       99.0       2000       1957       +/-       45.         ISOPROFYLMENEENE       106-41-51-8       99.0       2001       2022       +/-       28.         N-HYLLENEXENE       106-42-3       99.9       2001       2028       +/-       29.         N-BUTYLEENZENE       106-42-3       99.9       2000 <td< td=""><td>4 LA55</td></td<>	4 LA55
CIS 1, 3-DICHLOROPROPENE (Z) 10061-01-5 96.1 2000 2036 +/- 12. CIS-1, 2-DICHLOROFTHANE 156-59-2 97.6 2000 1947 +/- 26. DIEROMONENTAANE 124-48-1 99.9 2001 2022 +/- 11. DIEROMONENTAANE 74-95-3 99.8 2000 2000 +/- 33. ETHYLBENEENE 100-41-4 99.5 2000 2040 +/- 8. HEXACHLOROBUTADIENE 87-68-3 99.2 2001 1946 +/- 45. IGOROFYLBENZENE (CURENE) 98-82-8 99.0 2000 2012 +/- 17. M-XYLENE (5) 108-36-3 99.8 2001 ***** METHYLBENZENE 104-51-8 98.7 2000 1957 +/- 28. N-EDTYLBENZENE 104-51-8 99.7 2000 1956 +/- 39. O-XYLENE 100 FORTULEN 99-97 2001 2028 +/- 15. NAPHITHALENE 91-20-3 99.9 2001 2028 +/- 15. NAPHITHALENE 91-20-3 99.9 2001 2022 +/- 39. O-XYLENE 95-47-6 99.5 2000 1956 +/- 39. O-XYLENE 95-47-6 99.5 2000 1956 +/- 20. P-IGOROFYLTOLUENE 95-47-6 99.9 2001 1956 +/- 20. P-XILENE 5) 106-42-3 99.9 2001 1956 +/- 20. P-XILENE 135-98-8 99.4 2000 1993 +/- 31. STYRENE 100-42-5 99.9 2001 2022 +/- 20. INFERTINGENZENE 135-98-8 99.4 2000 1993 +/- 31. STYRENE 100-42-5 99.9 2001 2012 +/- 12. INFERT-BUTYLBENZENE 106-42-3 99.9 2001 2012 +/- 12. INFERT-BUTYLBENZENE 106-42-3 99.9 2001 2012 +/- 12. INFERT-BUTYLBENZENE 100-42-5 99.9 2001 2012 +/- 12. INFERT-BUTYLBENZENE 106-42-5 99.9 2001 2012 +/- 12. INFERT BUTYLBENZENE 106-42-6 90.9 2000 1981 +/- 21. INFERT BUTYLBENZENE 106-40 0000 1981 -/- 25.	3 LB09
CTS-1,2-DICHLORORETHTIENE       156-59-2       97.6       2000       1947       +/- 26.         DIERCMOCHLOROMETHANK       124-48-1       99.9       2001       2022       +/- 11.         DIERCMOCHLOROMETHANK       124-48-1       99.9       2000       2000       +/- 33.         FIRTHIBANE       100-41-4       99.5       2000       2040       +/- 45.         IBCROMONETHANK       100-41-4       99.9       2001       2440       +/- 45.         IBCROMONETHANKEN       100-41-4       99.9       2000       2012       +/- 45.         IBCROMONETHANKENE       100-41-4       99.9       2000       2012       +/- 45.         IBCROMONETHANKENE       106-61-3       99.8       2001       196       +/- 45.         IBCROMONETHANKENE       104-51-8       98.7       2000       1956       +/- 26.         N-BUTYLEENZENE       101-65-1       99.9       2000       1956       +/- 26.       -/- 29.         N-PROFYLEENZENE       91-20-3       99.9       2000       1966       +/- 20.       -/- 20.         P-ISOPROFUTURINENK       91-20-3       99.9       2000       1981       +/- 20.       -/- 20.         P-ISOPROFUTURINENKENE       106-4	8 LA55
DIBROWCHLOROMETHANE       124-48-1       99.9       2001       2022       +/-       11.         DIBROWCHLOROMETHANE       74-95-3       99.8       2000       2000       +/-       33.         ETHYLBENZENE       100-41-4       99.5       2000       2010       +/-       8.         HEXACHLOROMETADIENE       87-68-3       98.2       2001       1946       +/-       8.         ISOPROPYLENZENE (CUMENE)       98-82-8       99.0       2000       2012       +/-       17.         M-KILENE (5)       108-36-3       98.8       2001       1957       +/-       28.         N-BUTYLENENCENCORDE       75-09-2       99.9       2000       1956       +/-       25.         N-FROFVILENZENE       103-65-1       99.9       2001       2028       +/-       15.         N-PEROFVILENZENE       103-65-1       99.9       2000       1956       +/-       20.         O-XULENE       91-20-3       99.9       2000       1986       +/-       20.         P-ISOPFOFVIDUENE       91-20-3       99.9       2000       1986       +/-       20.         P-XULENE (5)       106-42-3       99.9       2000       1931       +/- </td <td>1 LA60</td>	1 LA60
DIBROMOMETHANE 74-95-3 99.8 2000 2000 +/- 33. FTHYLENENENE 100-41-4 99.5 2000 2010 +/- 8. HEXACHLOROBUTADIENE 87-68-3 98.2 2001 1946 +/- 45. INFORMETHANE (CUMENE) 98-82-8 99.0 2001 21 +/- 17. N-XYLENE (5) 108-38-3 99.8 2001 ***** METHYLENE CHLORIDE 75-09-2 99.9 2000 1957 +/- 28. N-BUTYLENEXENE 103-65-1 99.9 2001 2028 +/- 15. NAPHTHALENE 91-20-3 99.9 2000 1956 +/- 39. O-XYLENE 5) 106-42-3 99.9 2000 1956 +/- 39. O-XYLENE 5) 106-42-3 99.9 2000 1956 +/- 39. P-ISOFROFYLOLUENE 99-7-6 99.9 2000 1956 +/- 39. P-ISOFROFYLOLUENE 195-98-6 99.4 2000 1993 +/- 31. STYRENE 100-42-5 99.9 2001 2012 +/- 11. TERT-CHLOROFTHENE 100-42-5 99.9 2001 2012 +/- 11. TERT-CHLOROFTHENE 127-18-4 99.9 2001 2012 +/- 13. STYRENE 100-42-5 99.9 2001 3981 +/- 21. TERT-CHLOROFTHENE 127-18-4 99.9 2001 2029 +/- 29. (1) Listed in alphabetical order. (2) Determined by capillary GC-FTD, unless otherwise noted. (3) NIST traceable weights are used to verify balance calibration with the preparation of e Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 94% pure. (4) Determined by capillary GC-FTD, unless otherwise noted. (5) These products coelute and are not quantified in the final mix. MINENT supports of the information contained in this publication. Support Deughty Dualty Control Supervisor	7 LA97
Differentiation       11.12	2 LA87
<ul> <li>HINDLAND</li> <li>HIN</li></ul>	6 LA39
INCREMENTING (CUMENE)       98-82-8       99.0       2000       2012       +/-       17.         M-XYLENE (5)       108-38-3       99.8       2001       ******       ******         METHYLENE (CHORIDE       75-09-2       99.9       2000       1957       +/-       28.         N-BUTYLENEXENE       104-51-8       98.7       2000       1956       +/-       29.         N-PUTYLENEXENE       103-65-1       99.9       2000       1950       +/-       39.         O-XYLENE       91-20-3       99.9       2000       1956       +/-       20.         P-ISOPROFYLTOLUENE       99-87-6       99.9       2000       1966       +/-       20.         P-XYLENE (5)       106-42-3       99.9       2000       1986       +/-       20.         P-XYLENE (5)       106-42-3       99.9       2001       2012       +/-       11.         STYRENE       100-42-5       99.9       2001       1012       +/-       12.         TERT-BUTYLENEXENE       180-466       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       12       127-18-4       99.9       2001       2029 <td>0 LA40</td>	0 LA40
<ul> <li>N-XYLENE (5) 108-36-3 99.8 2001 *****</li> <li>METHYLENE CHLORIDE 75-09-2 99.9 2000 1957 +/- 28.</li> <li>N-BUTYLENZENE 104-51-8 98.7 2000 1956 +/- 25.</li> <li>N-PEOPYLENZENE 104-51-99.9 2000 1956 +/- 39.</li> <li>O-XYLENE 91-20-3 99.9 2000 1950 +/- 39.</li> <li>O-XYLENE 91-20-3 99.9 2000 1950 +/- 39.</li> <li>O-XYLENE 91-20-3 99.9 2000 1956 +/- 20.</li> <li>P-ISOPROFYLTOLUENE 99.87-6 99.9 2000 1986 +/- 20.</li> <li>P-ISOPROFYLTOLUENE 135-98-8 99.4 2000 1993 +/- 31.</li> <li>STYRENE 100-42-5 99.9 2001 2012 +/- 11.</li> <li>TERT-BUTYLENZENE 98-06-6 99.9 2001 2012 +/- 12.</li> <li>TERT-BUTYLENZENE 127-18-4 99.9 2001 2012 +/- 12.</li> <li>TERT-BUTYLENZENE 127-18-4 99.9 2001 2029 +/- 29.</li> <li>(1) Listed in alphabetical order.</li> <li>(2) Determined by capillary GC-FID, unless otherwise noted.</li> <li>(3) NIST traceable weights are used to verify balance calibration with the preparation of encontration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corected for analytes less than 9% pure.</li> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot replicate injections.</li> <li>(5) These products coclute and are not quantified in the final mix.</li> </ul>	0 LA95
<ul> <li>MATHYLENE CHLORIDE</li> <li>N-BUTYLBENZENE</li> <li>N-BUTYLBENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>NO-FROPYLENZENE</li> <li>SSC-BUTYLBENZENE</li> <li>SSC-BUTYLENZENE</li> <li>SSC-BUTYLBENZENE</li> <li>SSC-BUTHENE</li> <li>106-42-5</li> <li>SSC-BUTYLBENZENE</li> <li>SSC-BUTHENE</li> <li>107-18-4</li> <li>SSC-BUTYLBENZENE</li> <li>SSC-BUTHENE</li> <li>127-18-4</li> <li>SSC-BUTHENE</li> <li>128-10000</li> <li>128-1</li></ul>	3 LB01
N-BUTYLBENZENE       104-51-8       98.7       2001       1996       +/-       25.         N-PEOPYLBENZENE       103-65-1       99.9       2001       2028       +/-       15.         NAPHTHALENE       91-20-3       99.9       2000       1950       +/-       39.9         O-XYLENE       99-67-6       99.9       2000       1986       +/-       20.9         P-XIENE (5)       106-42-3       99.9       2000       *****         SEC-DUTYLENZENE       135-98-8       99.4       2000       1993       +/-       31.         STYRENE       100-42-5       99.9       2001       2012       +/-       13.         STYRENE       100-42-5       99.9       2001       2012       +/-       14.         TERT-BUTYLBENZENE       127-18-4       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary CC-FID, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of education is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.       (4)       Determined by chromatographic analysis against an independently prepared reference lot. r	LB15
N. PROPTLEENEENE       103-65-1       99.9       2001       2028       +/-       15.         NAPHTHALENE       91-20-3       99.9       2000       1950       +/-       39.         O-XYLENE       99-87-6       99.9       2000       1966       +/-       20.         P-ISOPROFYLTOLUENE       99-87-6       99.9       2000       1966       +/-       20.         P-XYLENE       106-42-3       99.9       2000       1966       +/-       20.         P-XYLENE       135-98-8       99.4       2000       1993       +/-       31.         STYPENE       100-42-5       99.9       2001       2012       +/-       12.         TERT-BUTHBENZENE       180-66       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       127-18-4       99.9       2001       2029       +/-       29.         (2)       Determined by capillary GC-FID, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of eaco concentration of analyte in solution is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.         (4)       Determined by chromatographic analys	9 LA88
NNPHTHALENE       91-20-3       99.9       2000       1950       +/-       39.9         O-XYLENE       95-47-6       99.5       2000       2022       +/-       9.9         P-ISOPROFULTOLUENE       99-87-6       99.9       2000       1986       +/-       20.0         P-XYLENE (5)       106-42-3       99.9       2000       1986       +/-       11.         SEC-DUTYLEENZENE       135-98-8       99.4       2000       1983       +/-       11.         TERT-BUTYLEENZENE       135-98-8       99.9       2001       2012       +/-       11.         TERT-BUTYLEENZENE       127-18-4       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary GC-FID, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of e concentration of analyte in solution is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.         (4)       Determined by chromatographic analysis against an independently prepared reference lot replicate injections.       (5)       These products coclute and are not quantified in the final mix.         Muality Control Supervisor       Determine the suitability of the product fo	3 LB09
O-XYLENE       95-47-6       99.5       2000       2022       +/-       9.         P-ISOPROFYLTOLUENE       99-87-6       99.9       2000       1986       +/-       20.         P-XYLENE (5)       106-42-3       99.9       2000       1993       +/-       31.         STYLENE       135-98-8       99.4       2000       1993       +/-       11.         STYLENE       100-42-5       99.9       2001       2012       +/-       12.         TERT-BUTYLBENZENE       98-06-6       99.9       2000       1981       +/-       21.         TERT-BUTYLBENZENE       127-18-4       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary GC-FD, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of econcentration of analyte in solution is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 96% pure.         (4)       Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.       (5)       These products colute and are not quantified in the final mix.         Mused       Doughty       Determine the suitability of the product for its particula	6 LA92
<ul> <li>P-ISOPROPTLTOLUENE 99.9 2000 1986 +/- 20.</li> <li>P-XYLENE (5) 106-42-3 99.9 2000 *****</li> <li>SSC-BUTYLBENZENE 135-98-8 99.4 2000 1993 +/- 31.</li> <li>STYRENE 100-42-5 99.9 2001 2012 +/- 11.</li> <li>TERT-BUTYLBENZENE 98-06-6 99.9 2000 1981 +/- 20.</li> <li>TETRACHLOROSTHEME 127-18-4 99.9 2001 2029 +/- 29.</li> <li>(1) Listed in alphabetical order.</li> <li>(2) Determined by capillary GC-FID, unless otherwise noted.</li> <li>(3) NIST traceable weights are used to verify balance calibration with the preparation of e Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coolute and are not quantified in the final mix.</li> </ul>	5 LA97
P-XTLENE (5)       106-42-3       99.9       2000       *****         SEC-BUTYLBENZENE       135-98-8       99.4       2000       1993       +/-31.         STTRENE       100-42-5       99.9       2001       2012       +/-11.         TERT-BUTYLBENZENE       98-06-6       99.9       2000       1981       +/-21.         TERT-BUTYLBENZENE       127-18-4       99.9       2001       2029       +/-29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary GC-FTD, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of endoconcentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.         (4)       Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.         (5)       These products coelute and are not quantified in the final mix.         Mused       Determine the suitability of the product for its paticular use. Please see the latest         Piloword Doughty       Determine the suitability of the product for its paticular use. Please see the latest         Delowarants that its products conform to the information contained in this publication. chaser must determine the suitability of the product for its paticular use. Please see the latest	8 LB08
<ul> <li>FAILENS (3)</li> <li>FOURT STREENENT (3)</li> <li>STORENE (3)<!--</td--><td>7 LA41</td></li></ul>	7 LA41
STYRENE       100-42-5       99.9       2001       2012       +/-       11.         TERT-BUTYLBENZENE       98-06-6       99.9       2000       1981       +/-       21.         TERT-BUTYLBENZENE       127-18-4       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary GC-FD, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of enconcentration of analyte in solution is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.         (4)       Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.       (5)       These products coelute and are not quantified in the final mix.         Mused       DateHTY       Determine the suitability of the product for its particular use. Please see the latest the precision of the additione torus and conditiones de the determines the suitability of the product for its particular use. Please see the latest the product so colduct or its particular use. Please see the latest the product of the product for its particular use. Please see the latest the product of the product for its particular use. Please see the latest the product of the product for its particular use. Please see the latest the product of the product for its particular use. Please see the latest the product of the product for its particular use. Please see the latest the product for its particular use. Please see the latest	LB04
TERT-BUTTLBENZENE       98-06-6       99.9       2000       1981       +/-       21.         TETRACHLOROETHENE       127-18-4       99.9       2001       2029       +/-       29.         (1)       Listed in alphabetical order.       (2)       Determined by capillary GC-FID, unless otherwise noted.       (3)       NIST traceable weights are used to verify balance calibration with the preparation of enduction is ug/ml +/-       0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.         (4)       Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.       (5)         (5)       These products coelute and are not quantified in the final mix.         Wood Doughty Ruality Control Supervisor       Eleo warrants that its products conform to the information contained in this publication. chaser must determine the suitability of the product for its particular use. Please see the latest Supervisor	6 LA51
<ul> <li>TETRACHLOROSTHENE 127-18-4 99.9 2001 2029 +/- 29.</li> <li>(1) Listed in alphabetical order.</li> <li>(2) Determined by capillary GC-FID, unless otherwise noted.</li> <li>(3) NIST traceable weights are used to verify balance calibration with the preparation of editors.</li> <li>(3) NIST traceable weights are corrected for analytes less than 98% pure.</li> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coelute and are not quantified in the final mix.</li> </ul>	8 LB09
<ol> <li>Listed in alphabetical order.</li> <li>Determined by capillary GC-FID, unless otherwise noted.</li> <li>NTST traceable weights are used to verify balance calibration with the preparation of end Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>These products coelute and are not quantified in the final mix.</li> </ol>	
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<ul> <li>(3) NIST traceable weights are used to verify balance calibration with the preparation of e Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>(4) betermined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coelute and are not quantified in the final mix.</li> </ul>	
<ul> <li>Concentration of analyte in solution is ug/ml +/- 0.5%, based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coelute and are not quantified in the final mix.</li> </ul>	
<ul> <li>volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coolute and are not quantified in the final mix.</li> </ul>	ach lot.
<ul> <li>(4) Determined by chromatographic analysis against an independently prepared reference lot. replicate injections.</li> <li>(5) These products coolute and are not quantified in the final mix.</li> <li>(4) Determine the source of the product of the product for the pr</li></ul>	
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Bellefonte, PA 1682 Phone(814)35	3-0048 USA

# Table 8. 54 Component Mixture

**Company Confidential & Proprietary** 

Certij	ficate o	f Аn	larys	515	11.1	J. PAGE	0 15-20 0 1-4 2 of 2
DESCRIPTION: 502/524 Volatile C	Organics Calibrat	tion Mix					
CATALOG NO.: 502111		MFG DATE:		Nov~2003			
LOT NO.: LB16275		EXPIRATIO	N DATE:	Mar-2006			
SOLVENT: METHANOL							
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)		) ANALYTICAL (4) CENTRATION	)	STD DEV	SUPELCO LOT NO
TOLUENE	108-88-3	99.7	2001	2020	+/-	15.8	LA90411
TRANS 1,3-DICHLOROPROPENE (E)	10061-02-6	98.5	2000	2052	+/-	12.9	LE06449
TRANS-1,2-DICHLOROETHYLENE	156-60-5	99.9	2000	1910	+/-	36.2	LB02428
TRICHLOROETHYLENE 1,1-DICHLOROETHANE	79-01-6	98.5	2001	1980	+/-	20.2	LB04303
1,1-DICHLOROETHANE	75-34-3 75-35-4	97.0 99.9	2000 2000	1968 1980	+/- +/-	32.1 46.1	LA54711 LB04593
1,1-DICHLOROPROPENE	563~58~6	98.0	2000	1958	+/-	20.8	LB12558
1,1,1-TRICHLOROETHANE	71-55-6	99.9	2000	1973	+/-	26.8	LB14220
1, 1, 1, 2-TETRACHLOROETHANE	630-20-6	99.1	2001	2000	+/	16.1	LB01555
1, 1, 2-TRICHLOROETHANE	79-00-5	99.3	2000	2038	+/	12.6	LB03464
1,1,2,2-TETRACHLOROETHANE	79-34-5	97.5	2000	1974	+/-	31.7	LA86969
1,2-DIBROMO-3-CHLOROPROPANE	96-12-B	97.9	2000	1978	+/-	43.5	LB06608
1,2-DIBROMOETHANE	106-93-4	99.6	2001	2029	+/-	0.1	LA87068
1,2-DICHLOROBENZENE	95-50-1	99.9	2000	2008	+/-	29.2	LA96474
1, 2-DICHLOROFTHANE	107-06-2	99.9 99.9	2000	1974	+/- +/~	25.7	LA88777 LB08115
1,2-DICHLOROPROPANE 1,2,3-TRICHLOROBENZENE	78-87-5 87-61-6	99.9 99.75	2000 2000	2019 1962	+/~	9.6 18.9	LA50762
1,2,3-TRICHLOROPROPANE	96-18-4	99.1	2000	2005	+/-	17,8	LA39379
1,2,4-TRICHLOROBENZENE	120-82-1	98.6	2000	1957	+/-	52.1	LB12944
1,2,4-TRIMETHYLBENZENE	95-63-6	98.2	2000	2000	+/-	22.0	LA39081
1,3-DICHLOROBENZENE,	541-73-1	99.9	2001	2013	+/-	16.7	LA72024
1, 3-DI CHLOROPROPANE	142-28-9	99.9	2000	2024	+/-	11.8	LB00875
1,3,5-TRIMETHYLBENZENE	108-67-8	99.0	2000	2011	+/-	13.6	LA94493
1,4-DICHLOROBENZENE	106-46-7	99.9	2000	1992	+/-	16.2	LA50188
2-CHLOROTOLUENE	95~49~8	99.9	2000	2005	+/-	23.6	LA95842
2,2-DICHLOROPROPANE	594-20-7	98.3	2000	1968	+/-	19.4	LB01750
4-CHLOROTOLUENE	106-43-4	99.9	2001	1990	+/-	15.0	LB05252
<ul><li>(1) Listed in alphabetical order.</li><li>(2) Determined by capillary GC-FII</li></ul>	unless other	ise notod					
<ul><li>(2) Determined by capitlary GC-Fit</li><li>(3) NIST traceable weights are use</li></ul>			ration wi	th the prepara	tion	of each	ılot.
Concentration of analyte in so							
volumetric glassware. Weights							
(4) Determined by chromatographic		-		-	ence	lot. Me	ean of
replicate injections.							
(5) These products coelute and are	e not quantified	in the fir	nal mix.				
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wood Doughty uality Control Supervisor							
elco warrants that its products conform to the inform	ation contained in th	is publication		Gc		DFI	CO
haser must determine the suitability of the product for its	particular use. Please	see the lates	t	S .	(البينة م	l less la	a and 2007
og or order invoice and packing slip for additional terms				50	15North	Harrison R	oad

## Table 9. 8260 + Mix

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222	12753	<u>.</u>	<b>633</b>	69a	67
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		anis I	633	Rez V	E

11 - XX	Masc 5	
Chemical Stan	dard Batch Sheet	

Lot #: A042263

Catalog #: 552504A	Target: 1000 - 40000 ug/ml							
Description: Custom Vola	tiles Standard N	fix A						
Solvent: P&T Methan	ol	Solvent	Lot: 44337			Final Volum	e: 100	ml
								· · · · · · · · · · · · · · · · · · ·
Made by: Joe Tallon			Date: 1/4/2	2006 8:09	9:50A			
Tested by:			Date:					
			By:	····		Date:		
Packaged by: Jackie Glass	gow / Staci Bod	le	Date: 1/4/2	2006 10:4	9:12/	No. Units:	12	
Balance Used: AT261			Serial #: 1119	9141429				
		Storage			Target	Target	Actual	Calc
Compound	CAS	<u>Location</u>	<u>Lot #</u>	Purity	Conc(ug/ml)	<u>Weight</u>	Weight	Conc(ug/ml)
Carbon disulfide	75-15-0	FA1A5D	J11J02	0.99	1,000.00	100.00	100.00	1,000.00
Methyl-tert-butyl ether (	1634-04-4	FA1B6C	10660BD	0.97	1,000.00	100.00	100.00	1,000.00
Iodomethane (methyl	74-88-4	FA1C2A	13906AB	0.99	1,000.00	100.00	100.00	1,000.00
Ethyl methacrylate	97-63-2	FA1C1D	09316HC	0.99	1,000.00	100.00	100.00	1,000.00
Tetrahydrofuran	109-99-9	FA1B8B	01057MC	0.99	5,000.00	500.00	500.00	5,000.00
trans-1,4-dichloro-2-butene	110-57-6	FA1C1C	160-22DD	0.99	5,000.00	500.00	500.00	5,000.00
Acetonitrile	75-05-8	FA1B13A	12067KC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FAIA11A	01404PV	0.99	1,000.00	100.00	100.00	1,000.00
Methyl acetate	79-20-9	FAICIIC	47640/1	0.99	1,000.00	100.00	100.00	1,000.00
Methylcyclohexane	108-87-2	FA1E4A	02759BC	0.99	1,000.00	100.00	100.00	1,000.00
Cyclohexane	110-82-7	FA1C7A	03145KB	0.99	1,000.00	100.00	100.00	1,000.00



8260 H 13 WSC5 11-720 Chemical Standard Batch Sheet

Lot #: A042264

Catalog #: 552504B		Target: 5000 ug	z/ml			~····		
Description: Custom Volat	iles Standard M	lix B						
Solvent: P&T Methand	ol 🛛	Solvent ]	Lot: A041266			Final Volume	e: 50	ml
Made by: Joe Tallon		······································	Date: 1/4/2	006 8:21	)-50 A			
Tested by:			Date:	.000 8.30	7			
Tested by:								
			By:			Date:		
Packaged by: Jackie Glasg	ow / Staci Bodl	e	Date: 1/4/2006 10:54:16/ No. Units: 12					
Balance Used: AT261			Serial #: 1119	141429				
		<u>Storage</u>			<u>Target</u>	<u>Target</u>	<u>Actual</u>	Calc
Compound	CAS	Location	<u>Lot #</u>	<u>Purity</u>	Conc(ug/ml)	<u>Weight</u>	Weight	Conc(ug/ml)
2-Chloroethyl vinyl ether	110-75-8	FAIA11D	03206CI	0.99	5,000.00	250.00	250.00	5,000.00

	DESCRIPTION: SEVERN	TRENT LABS	M	<i>ition</i> इ.५७ vsc ४२ ५	-> 13
	QUOTE 20460869	LOT NO.; LB2	:5705	MFG DATE: Dec-2004	
	SOLVENT: DEIONIZED WAY	TER			
_	ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
	ACROLEIN ACRYLONITRILE	107-02-8 107-13-1	98.4 99.9	20008 +/- 100 20000 +/- 100	
			·		
	<ol> <li>Listed in alphabetical order.</li> <li>Determined by capillary GC-FII</li> <li>NIST traceable weights are use Concentration of analyte in soc Class A volumetric classers</li> </ol>	d to verify balanc lution is ug/ml +/	e calibration wit - 0.5%, uncertain	ity based upon balance	each lot. and
	Class A volumetric glassware.	Mergnes are corre	cced for analytes	iess than 98% pure.	
	7. Doncebre				
A N elo	bod Doughty Manager powarrants that its products conform to the info aser must determine the suitability of the product for g or order invoice and packing slip for additional te	its narticular use. Please	e see the latest		ELCO Road • Bellaforita, PA Phone (814) 359-3441



8240+#4 mvsc 5 1-710

Chemical Standard Batch Sheet Lot #: A042268

Catalog #: 55684	3	Target: 5000 u	g/ınl					· · · · · · · · · · · · · · · · · · ·
Description: Custor	n Vinyl Acetate Standa	ırd						······
Solvent: P&T N	Methanol	Solvent	Lot: A038421	••••••••		Final Volume	25	ml
Made by: Joe 7	allon	r	Date: 1/4/2	2006 9:41	D:21A		····	
Tested by:			Date:					
	······································	<u> </u>	By:	· · ·		Date:		
Packaged by: Jacki	e Glasgow / Staci Bod	e	Date: 1/4/2	006 10:5	8:29/	No. Units:	12	
Balance Used: AT20	Balance Used: AT261			141429				
·····		Storage	1		Target	Target	Actual	Calc
ompound	CAS	Location	<u>Lot #</u>	<u>Purity</u>	Conc(ug/ml)	Weight	Weight	Conc(ug/ml
inyl acetate	108-05-4	FA1A9A	08831CW	0.99	5,000.00	125.00	125.00	5,000.00

SOP No. BF-MV-005, Rev. 2 Effective Date: 03/30/2010 Page No.: 46 of 58 221T



MUSC 23 6-720 24 [-75 Gravimetric Certificate

P^llefonte, PA Tel: (800)3	Tel: (800)356-1688 Catalog No.		tom Ketones Standard	Lot No.: <u>A044128</u>	R TO USE.
Component #	Compound	CAS#	Percent Purity <sup>2</sup>	Concentration <sub>3</sub> (weight/volume) <sup>3</sup>	Percent Uncertainty <sup>4</sup>
1	2-Butanone (MEK)	78-	93-3 99%	5,000.00 ug/ml	+/-0.08 %
2	2-Hexanone	591-	78-6 99%	5,000.00 ug/m1	+/-0.08 %
3	4-Methyl-2-pentanone (MIBK)	108-	10-1 99%	5,000.00 ug/ml	+/~0.08 %
4	Acetone	67-0	54-1 99%	5,000.00 ug/ml	+/-0.08 %
Solvent:	P/T Methanol/Water (90:10)				

 F. Joseph Jallon - Mix Technidan
 Balance: 1119141429

 F. Joseph Jallon - Mix Technidan
 Balance: 1119141429

 F. Twition date of the unopened ampul stored at recommended temperature.
 was determined by one or more of the following techniques: BC#FID, HPLC, GC/ECD, GCMS, Value rounded to using 1 or more of the following: MS, DSC, solid probe MS, GC/FD, BONPD, GC/TC, FIR, melling point, refaceive index, and Karl Fisher. See data pack or contact Resek for further details.

 Based upon gravimetic preperation with balance calibration verified using NSTraceable weights (seven rracs) evels).

 iPercent Uncertainty based upon balance AND ASTM Class Avolumetric glassware accuracy.



#### Table 10. Second Source 60 Component Mixture

SCIENTIFIC Analytical Solutions	Certificate of Analysis	ę.	S. Sourcie
	VOC Mixture		

Product DWM-588 Lot Number: CB-2659

Expiration Date: Dec-2008 Page:

1 of 3

This Certified Reference Material (CRM) was manufactured and verified in accordance with ULTRA's ISO 9001:2000 registered quality system, and the analyte concentrations were verified by our ISO 17025 accredited laboratory. The true value and uncertainty value at the 95% confidence level for each analyte, determined gravimetrically, is listed below.

bromochloromethane $000074-97-5$ JS-16015HS $i$ $2006 \pm 10 \ \mu g/s$ bromodichloromethane $000075-27-4$ DU-14522LS $2006 \pm 10 \ \mu g/s$ bromoform $000075-25-2$ DU-06126KS $2006 \pm 10 \ \mu g/s$ carbon tetrachloride $000056-23-5$ $01704MF$ $2006 \pm 10 \ \mu g/s$ chloroform $000067-66-3$ BS-03041BS $2006 \pm 10 \ \mu g/s$ dibromochloromethane $000124-48-1$ DO-12622CI $2006 \pm 10 \ \mu g/s$ dibromomethane $000075-09-2$ $44267$ $2006 \pm 10 \ \mu g/s$ methylene chloride $000075-69-4$ DR-16417BR $2006 \pm 10 \ \mu g/s$ 1,2-dibromoethane $000075-69-4$ DR-16417BR $2006 \pm 10 \ \mu g/s$ 1,2-dichloroethane $000075-34-3$ $64552/1$ $2006 \pm 10 \ \mu g/s$ 1,1-dichloroethane $000075-35-4$ $01218EC$ $2007 \pm 10 \ \mu g/s$ 1,1-dichloroethane $000075-35-4$ $01218EC$ $2006 \pm 10 \ \mu g/s$ 1,1-dichloroethene $000156-59-2$ $13707BO$ $2006 \pm 10 \ \mu g/s$ 1,1,4,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,4,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \ \mu g/s$ 1,1,2,2-te	Analyte	CAS#	Analyte Lot	True Value
bromoform000075-25-2DU-06126KS2006 $\pm 10 \mu g/t$ carbon tetrachloride000056-23-501704MF2006 $\pm 10 \mu g/t$ chloroform000067-66-3BS-03041BS2006 $\pm 10 \mu g/t$ dibromochloromethane000124-48-1DO-12622Cl2006 $\pm 10 \mu g/t$ dibromochloromethane000075-09-2442672006 $\pm 10 \mu g/t$ methylene chloride000075-69-4DR-16417BR2006 $\pm 10 \mu g/t$ trichlorofluoromethane000075-69-4DR-16417BR2006 $\pm 10 \mu g/t$ 1,2-dibromoethane000075-34-364552/12006 $\pm 10 \mu g/t$ 1,1-dichloroethane000075-34-364552/12006 $\pm 10 \mu g/t$ 1,1-dichloroethane000075-35-401218EC2007 $\pm 10 \mu g/t$ 1,1-dichloroethene000156-59-213707BO2006 $\pm 10 \mu g/t$ 1,1,4,2-tetrachloroethane0000630-20-6CO-12312Ll2006 $\pm 10 \mu g/t$ 1,1,4,2-tetrachloroethane000075-34-510917TB2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000075-34-6CO-12312Ll2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000075-34-510917TB2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000075-34-510917TB2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000075-34-510917TB2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000176-34-510917TB2006 $\pm 10 \mu g/t$ 1,1,2,2-tetrachloroethane000176-34-510917T	•	000074-97-5		2006 ± 10 µg/mL
carbon tetrachloride000056-23-501704MF2006 $\pm$ 10 µg/hchloroform000067-66-3BS-03041BS2006 $\pm$ 10 µg/hdibromochloromethane000124-48-1DO-12622CI2006 $\pm$ 10 µg/hdibromomethane000074-95-3EM-01514TJ2006 $\pm$ 10 µg/hdibromomethane000075-09-2442672006 $\pm$ 10 µg/hmethylene chloride000075-69-4DR-16417BR2006 $\pm$ 10 µg/htrichlorofluoromethane000075-69-4DR-16417BR2006 $\pm$ 10 µg/h1,2-dibromoethane000075-34-364552/12006 $\pm$ 10 µg/h1,1-dichloroethane000075-35-401218EC2007 $\pm$ 10 µg/h1,1-dichloroethene000156-59-213707BO2006 $\pm$ 10 µg/h1,1,1-dichloroethene000156-60-5DO-07817JR2006 $\pm$ 10 µg/h1,1,4,2-tetrachloroethane000063-20-6CO-12312LI2006 $\pm$ 10 µg/h1,1,2,2-tetrachloroethane000079-34-510917TB2006 $\pm$ 10 µg/h1,1,2,2-tetrachloroethane000079-34-510917TB2006 $\pm$ 10 µg/h1,1,2,2-tetrachloroethane000079-34-510917TB2006 $\pm$ 10 µg/h1,1,2,2-tetrachloroethane000079-34-510917TB2006 $\pm$ 10 µg/h1,1,2,2-tetrachloroethane000172-78-4PS-00344BR2006 $\pm$ 10 µg/h	bromodichloromethane	000075-27-4	DU-14522LS	2006 ± 10 μg/mL
chloroform         000067-66-3         BS-03041BS         2006 ± 10 µg/4           dibromochloromethane         000124-48-1         DO-12622Cl         2006 ± 10 µg/4           dibromomethane         000074-95-3         EM-01514TJ         2006 ± 10 µg/4           methylene chloride         000075-09-2         44267         2006 ± 10 µg/4           trichlorofluoromethane         000075-69-4         DR-16417BR         2006 ± 10 µg/4           1,2-dibromoethane         000075-69-4         DR-16417BR         2006 ± 10 µg/4           1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/4           1,2-dichloroethane         000107-06-2         KN-09446KN         2006 ± 10 µg/4           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/4           1,1-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/4           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/4           1,1,4,2-tetrachloroethane         000063-20-6         CO-12312LI         2006 ± 10 µg/4           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/4           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/4           1,1,2,2	bromoform	000075-25-2	DU-06126KS	2006 ± 10 µg/mL
dibromochloromethane $000124-48-1$ $DO-12622CI$ $2006 \pm 10 \mu gh$ dibromomethane $000074-95-3$ $EM-01514TJ$ $2006 \pm 10 \mu gh$ methylene chloride $000075-09-2$ $44267$ $2006 \pm 10 \mu gh$ trichlorofluoromethane $000075-69-4$ $DR-16417BR$ $2006 \pm 10 \mu gh$ 1,2-dibromoethane $000075-69-4$ $DR-16417BR$ $2006 \pm 10 \mu gh$ 1,2-dibromoethane $000075-34-3$ $64552/1$ $2006 \pm 10 \mu gh$ 1,1-dichloroethane $000075-34-3$ $64552/1$ $2006 \pm 10 \mu gh$ 1,2-dichloroethane $000075-35-4$ $01218EC$ $2007 \pm 10 \mu gh$ 1,1-dichloroethene $000075-35-4$ $01218EC$ $2006 \pm 10 \mu gh$ 1,1-dichloroethene $000156-59-2$ $13707BO$ $2006 \pm 10 \mu gh$ 1,1,2-dichloroethene $000156-60-5$ $DO-07817JR$ $2006 \pm 10 \mu gh$ 1,1,4,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \mu gh$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \mu gh$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \mu gh$ 1,1,2,2-tetrachloroethane $000079-34-5$ $10917TB$ $2006 \pm 10 \mu gh$	carbon tetrachloride	000056-23-5	01704MF	2006 ± 10 µg/mL
dibromomethane         000074-95-3         EM-01514TJ         2006 ± 10 µg/n           methylene chloride         000075-09-2         44267         2006 ± 10 µg/n           trichlorofluoromethane         000075-69-4         DR-16417BR         2006 ± 10 µg/n           1,2-dibromoethane         000075-69-4         DR-16417BR         2006 ± 10 µg/n           1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/n           1,2-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/n           1,1-dichloroethene         000075-35-4         01218EC         2007 ± 10 µg/n           cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/n           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/n           1,1,4,2-tetrachloroethane         0000630-20-6         CO-12312LI         2006 ± 10 µg/n           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/n           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/n	chloroform	000067-66-3	BS-03041BS	2006 ± 10 µg/mL
methylene chloride         000075-09-2         44267         2006 ± 10 µg/n           trichlorofluoromethane         000075-69-4         DR-16417BR         2006 ± 10 µg/n           1,2-dibromoethane         000106-93-4         TB-101777         2006 ± 10 µg/n           1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/n           1,2-dichloroethane         000075-35-4         64552/1         2006 ± 10 µg/n           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/n           1,1-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/n           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/n           trans-1,2-dichloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/n           1,1,4,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/n           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/n	dibromochloromethane	000124-48-1	DO-12622CI	2006 ± 10 µg/mL
trichlorofluoromethane         000075-69-4         DR-16417BR         2006 ± 10 µg/h           1,2-dibromoethane         000106-93-4         TB-101777         2006 ± 10 µg/h           1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/h           1,2-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/h           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/h           1,1-dichloroethene         0000156-59-2         13707BO         2006 ± 10 µg/h           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/h           trans-1,2-dichloroethene         000630-20-6         CO-12312LI         2006 ± 10 µg/h           1,1,4,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/h           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/h           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/h	dibromomethane	000074-95-3	EM-01514TJ	2006 ± 10 µg/mL
1,2-dibromoethane         000106-93-4         TB-101777         2006 ± 10 µg/h           1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/h           1,2-dichloroethane         000107-06-2         KN-09446KN         2006 ± 10 µg/h           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/h           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/h           cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/h           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/h           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/h           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/h           1,1,2,2-tetrachloroethane         0000179-34-5         10917TB         2006 ± 10 µg/h           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/h	methylene chloride	000075-09-2	44267	2006 $\pm$ 10 µg/mL
1,1-dichloroethane         000075-34-3         64552/1         2006 ± 10 µg/n           1,2-dichloroethane         000107-06-2         KN-09446KN         2006 ± 10 µg/n           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/n           1,1-dichloroethane         000075-35-4         01218EC         2006 ± 10 µg/n           cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/n           trans-1,2-dichloroethane         000156-60-5         DO-07817JR         2006 ± 10 µg/n           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/n           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/n           1,1,2,2-tetrachloroethane         0000179-34-5         10917TB         2006 ± 10 µg/n           tetrachloroethene         000179-34-5         10917TB         2006 ± 10 µg/n	trichlorofluoromethane	000075-69-4	DR-16417BR	2006 ± 10 µg/mL
1,2-dichloroethane         000107-06-2         KN-09446KN         2006 ± 10 µg/r           1,1-dichloroethane         000075-35-4         01218EC         2007 ± 10 µg/r           cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/r           trans-1,2-dichloroethene         000156-69-2         13707BO         2006 ± 10 µg/r           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/r           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/r           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/r	1,2-dibromoethane	000106-93-4	TB-101777	2006 ± 10 µg/mL
1,1-dichloroethene         000075-35-4         01218EC         2007 ± 10 µg/r           cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/r           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/r           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/r           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/r	1,1-dichloroethane	000075-34-3	64552/1	2006 ± 10 µg/mL
cis-1,2-dichloroethene         000156-59-2         13707BO         2006 ± 10 µg/r           trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/r           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/r           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/r	1,2-dichloroethane	000107-06-2	KN-09446KN	2006 ± 10 µg/mL
trans-1,2-dichloroethene         000156-60-5         DO-07817JR         2006 ± 10 µg/r           1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/r           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         0000127-18-4         PS-00344BR         2006 ± 10 µg/r	1,1-dichloroethene	000075-35-4	01218EC	2007 ± 10 µg/mL
1,1,4,2-tetrachloroethane         000630-20-6         CO-12312LI         2006 ± 10 µg/r           1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/r	cis-1,2-dichloroethene	000156-59-2	13707BO	2006 ± 10 µg/mL
1,1,2,2-tetrachloroethane         000079-34-5         10917TB         2006 ± 10 µg/r           tetrachloroethene         000127-18-4         PS-00344BR         2006 ± 10 µg/r	trans-1,2-dichloroethene	000156-60-5	DO-07817JR	2006 ± 10 µg/mL
tetrachloroethene 000127-18-4 PS-00344BR 2006 ± 10 µg/r	1,1,4,2-tetrachloroethane	000630-20-6	CO-12312LI	2006 ± 10 µg/mL
	1,1,2,2-tetrachloroethane	000079-34-5	10917TB	2006 ± 10 µg/mL
1.1.4-trichloroethane 000071-55-6 LU-13149TR 2006 ± 10 µg/r	tetrachloroethene	000127-18-4	PS-00344BR	2006 ± 10 µg/mL
	1,1,1-trichioroethane	000071-55-6	LU-13149TR	2006 ± 10 µg/mL.
1,1,2-trichloroethane 000079-00-5 JB-0701HH 2006 ± 10 µg/r	1,1,2-trichloroethane	000079-00-5	JB-0701HH	2006 ± 10 µg/mL
trichloroethene 000079-01-6 KN-08846KN 2006 ± 10 µg/r	trichloroethene	000079-01-6	KN-08846KN	2006 $\pm$ 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.



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Dr. Edward Fitzgerald, Senior Scientist



# Certificate of Analysis

#### **VOC Mixture**

Product Lot Number:	DWM-588 CB-2659			Expiration Date:Dec-2008Page:2 of 3
Analyte		CAS#	Analyte Lot	True Value
1,2-dibromo-3-chl	oropropane	000096-12-8	OGF-01	2005 ± 10 µg/mL
1,2-dichloropropar	ne	000078-87-5	DC-120777	2005 ± 10 µg/mL
1,3-dichloropropar	ıe	000142-28-9	PR-17916MR	2006 ± 10 µg/mL
2,2-dichloropropar	ne	000594-20-7	CI-05304BI	2005 ± 10 μg/mL
1,1-dichloroproper	ne	000563-58-6	34768-21	2006 ± 10 µg/mL
cis-1,3-dichleropro	pene	010061-01-5	35072-03	2006 ± 10 µg/mL
trans-1,3-dichlorop	propene	010061-02-6	34251-41	2005 ± 10 µg/mL
hexachlorobutadie	ene	000087-68-3	339923/1	2005 $\pm$ 10 µg/mL
1,2,3-trichloroprop	ane	000096-18-4	12020TF	2006 ± 10 µg/mL
naphthalene		000091-20-3	14205KB	2005 ± 10 µg/mL
benzene		000071-43-2	31072	2006 ± 10 μg/mL
n-butylbenzene		000104-51-8	AA-28519CO	2005 ± 10 µg/mL
sec-butylbenzene		000135-98-8	MR-11305DN	2006 ± 10 µg/mL
tert-butylbenzene		000098-06-6	MQ-04010MQ	2006 ± 10 µg/mL
ethylbenzene		000100-41-4	033067	2005 ± 10 µg/ml.
isopropylbenzene		000098-82-8	EN-00621TG	2006 ± 10 µg/mL
4-isopropyltoluene		000099-87-6	PP-05104CP	2006 ± 10 µg/mL
n-propylbenzene		000103-65-1	LO-14503MR	2006 ± 10 µg/mL
styrene		000100-42-5	MQ-11229MQ	2005 ± 10 µg/mL
toluene		000108-88-3	43045	2006 ± 10 µg/mL
1,2,4-trimethylbenz	zene	000095-63-6	BO-13528BI	2006 ± 10 µg/mL
1,3,5-trimethylbenz	zene	000108-67-8	KM-02011HM	2007 ± 10 µg/mL
o-xylene		000095-47-6	DO-06834CO	2006 ± 10 µg/mL
m-xylene		000108-38-3	DI-00459CJ	2006 ± 10 µg/mL

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.



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Dr. Edward Fitzgerald, Senior Scientist

Expiration Date: Dec-2008



# Certificate of Analysis

#### VOC Mixture

Product DWM-588

Lot Number: CB-2659			Page: 3 of 3
Analyte	CAS#	Analyte Lot	True Value
p-xylene	000106-42-3	03747LN	2005 ± 10 µg/mL
1,4-dichlorobenzene	000106-46-7	06205KA	2005 ± 10 µg/mL
bromobenzene	000108-86-1	CG-02513MF	2006 ± 10 µg/mL
chlorobenzene	000108-90-7	63148HZ	2006 ± 10 µg/mL
2-chlorotoluene	000095-49-8	KS-06506BN	2005 ± 10 µg/mL
4-chlorotoluene	000106-43-4	CR-14512LQ	2005 ± 10 µg/mL
1,2-dichlorobenzene	000095-50-1	08946KY	2005 ± 10 µg/mL
1,3-dichlorobenzene	000541-73-1	JN-05902LZ	2006 ± 10 µg/mL
1,2,3-trichlorobenzene	000087-61-6	LI-12912PF	2006 ± 10 µg/mL
1,2,4-trichlorobenzene	000120-82 <b>-1</b>	00334TQ	2006 ± 10 µg/mL
bromomethane	000074-83-9	06623AQ	2008 ± 10 µg/mL
chloroethane	000075-00-3	00223KG	2009 ± 10 µg/mL
chloromethane	000074-87-3	07-44048	2009 ± 10 µg/mL
dichlorodifluoromethane	000075-71-8	N960053	2008 ± 10 µg/mL
vinyl chloride	000075-01-4	UN-1086	2009 ± 10 µg/mL
Matrix: methanol (methyl alcohol)			

Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.



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Dr. Edward Fitzgerald, Senior Scientist

# Table11. Second Source 8260 + Mixture

DESCRIPTION: SEVER	N TRENT LARS	-	82 Osition	VSC61-7
QUOTE 20687608	LOT NO.: LB3			
SOLVENT: METHANOL	HOI NO.: 1183	5787 EXPI	RATION DATE: Jan-2007	ſ
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3	SUPELCO ) LOT NO
ETONITRILE	75-05-8			
RBON DISULFIDE	75-15-0	99.9 99.9 (a)	40001 +/- 200. 999 +/- 5.	
CLOHEXANE	. 110-82-7	99.9	999 +/- 5. 1000 +/- 5.	
HYL METHACRYLATE	97-63-2	99.3	1000 +/- 5.	
EON 113	76-13-1	99.9 (b)	1001 +/- 5.	
THYL ACETATE	79-20-9	98.1	1001 +/- 5.	
THYL CYCLOHEXANE	108-87-2	99.8	1001 +/- 5.	
THYL TERT-BUTYL ETHER	1634-04-4	99.9	1002 +/- 5.	
IRAHYDROFURAN	109-99-9	97.4	4999 +/- 25.0	
ANS-1,4-DICHLORO-2-BUTENE	110-57-6	98.2	5002 +/- 25.0	D LB10202
CHLOROHEXANE	544-10-5	99.9	1000 +/~ 5.0	LB18907
<ol> <li>Listed in alphabetical order</li> <li>Determined by capillary GC-F</li> <li>a) GC; detector FPD</li> <li>b) GC; detector HALL</li> <li>NIST traceable weights are u Concentration of analyte in Class A volumetric glassware</li> </ol>	"ID, unless otherwise used to verify balance solution is ug/ml +/-	calibration wit	ity based upon balanc	each lot. e and

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Certif	ficate o	f Compo	osition	-020	SUL SOUT
DESCRIPTION: SEVERN TRE	NT LABS				11-72
QUOTE 20687609	LOT NO.: LB	35788 EXPI	RATION DATE: J	an-2007	
SOLVENT: DEIONIZED WATER METHANOL		50 % 50 %			
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGH CONCENTRA		SUPELCO LOT NO
ACETONE	67-64-1	99.9	5004 +.	/- 25.0	LB31953
IODOMETHANE	74-88-4	99.9		/- 5.0	LA73149
VINYL ACETATE	108-05-4	99.9	-	/- 25.0	LB31606
2-BUTANONE	78-93-3	99.9	5004 +,	/- 25.0	LB19842
2-HEXANONE 4-METHYL-2-PENTANONE	591~78-6 108-10-1	99.9 99.9	-	/- 25.0 /- 25.0	LB08447 LA99226
<ol> <li>Listed in alphabetical order.</li> <li>Determined by capillary GC-FID,</li> <li>NIST traceable weights are used Concentration of analyte in solu Class A volumetric glassware. Weights and the second second</li></ol>	to verify balan tion is ug/ml +	ce calibration wi /- 0.5%, uncertai	nty based upon	balance	ach lot. and
					-
Elwood Doughty QA Manager				Gen	PELCO
Supelco warrants that its products conform to the in Purchaser must determine the suitability of the product f catalog or order invoice and packing slip for additional	or its particular use. I	Please see the latest	5	95 North Harri	<b>РЕЦСО</b> ison Road • Bellefonte, PA A • Phone (814) 359-3441

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HEARDING: Y. CHURCHY, WINJ: HEART         MARKAN BARANANANANANANANANANANANANANANANANANANA	Ceri	ificate of Analysis	
<text><text><text><text><text><list-item></list-item></text></text></text></text></text>		Musc 66 3-7	
	DESCRIPTION: 2-Chloroethyl v	nyl ether	
OBJECTIVE METHOD       MARINE METHOD       MARINE METHOD       MARINE	CATALOG NO.: 40017	MFG DATE: Feb-2005	
	LOT NO.: LB27794	EXPIRATION DATE: Feb-2008	
NUMBER     PUBLICY (1)     CONCENTRATION     DEV     LOT M       2-CHLOROETHYL VINYL ETHER     110-75-8     99.9     5000     \$000     \$	SOLVENT: METHANOL		
<ol> <li>Betermined by capillary GC-FID, unless otherwise noted.</li> <li>NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is u/a/u i/- 0.5%, uncertainty based upon balance and class A volumetric glassware. Weights are corrected for analytes less than 98% pure.</li> <li>Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ol>	ANALYTE		
<ul> <li>(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.</li> <li>(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ul>	2-CHLOROETHYL VINYL ETHER	110-75-8 99.9 5000 5000 +/- 55.9 LE	101239
<ul> <li>(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.</li> <li>(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ul>			
<ul> <li>(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.</li> <li>(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ul>			
<ul> <li>(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.</li> <li>(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ul>			
<ul> <li>(2) NIST traceable weights are used to verify balance calibration with the preparation of each lot. Concentration of analyte in solution is ug/ml +/- 0.5%, uncertainty based upon balance and Class A volumetric glassware. Weights are corrected for analytes less than 9% pure.</li> <li>(3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections.</li> </ul>			
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Class A volumetric glassware. Weights are corrected for analytes less than 98% pure. (3) Determined by chromatographic analysis against an independently prepared reference lot. Mean of replicate injections. Wood Doughty uality Control Supervisor eloo warrants that its products conform to the information contained in this publication. these must determine the suitability of the product for its particular use. Please see the latest bog or order invoice and packing slip for additional terms and conditions of sale.	(2) NIST traceable weights are a	used to verify balance calibration with the preparation of each lo	t.
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elco warrants that its products conform to the information contained in this publication. haser must determine the suitability of the product for its particular use. Please see the latest log or order invoice and packing slip for additional terms and conditions of sale.	Iwood Doughty Quality Control Supervisor		
log or order invoice and packing slip for additional terms and conditions of sale. 595 North Harrison Road	elco warrants that its products conform to the ir	iormation contained in this publication.	0
Phone (814) 359-3441		erms and conditions of sale. 595 North Harrison Road Bellefonte, PA 16823-0048 U	

SOP No. BF-MV-005, Rev. 2 Effective Date: 03/30/2010 Page No.: 53 of 58 221T

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Cert	ificate o	f Comp	osition 824	Sec. Sorra MVSC7 1-710
i de la companya de l				MUSC7
DESCRIPTION: SEVERN				1-410
QUOTE 20687606		35789 EXPI	RATION DATE: Jul-2006	
SOLVENT: DEIONIZED WAY				
ANALYTE (1)	CAS NUMBER	PERCENT PURITY (2)	WEIGHT CONCENTRATION (3)	SUPELCO LOT NO
CROLEIN	107-02-8	98.4	00010 / 160 1	×704530
CRYLONITRILE	107-13-1	98.4 99.9	20012 +/- 100.1 20008 +/- 100.0	LB21530 LB25800
<ol> <li>Listed in alphabetical order.</li> <li>Determined by capillary GC-FI</li> </ol>	·····			
(3) NIST traceable weights are us	ed to verify balan	ce calibration wi		
Concentration of analyte in s Class A volumetric glassware.				and
5/1				
/wood WanGetty				
Elwood Doughty DA Manager			<b>S</b> SI	IPELCO
upelco warrants that its products conform to th urchaser must determine the suitability of the produ atalog or order invoice and packing slip for additic	uct for its particular use	Please see the latest	595NorthHa	rison Road • Bellefonta, PA SA • Phone (B14) 359-3441

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#### Table 12. 8260 Add Mixture

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P				K	

mvsC 71-18-20 72-01-07 Chemical Standard Batch Sheet Lot #: A042005

					1000 1110	2005		
Catalog #: 552546		Target: 2000-8	0000 ug/ml					
ription: Custom Vola	tiles Standard							
Solvent: P&T Methan		Solvent	Lot: 44337			Final Volume	: 100	ml
Made by: Ryan Miller	r		Date: 12/1	9/2005 10	0:12:4			
Tested by:		~	Date:					
		1	By:			Date:		
Packaged by: /	LBT.	Ja	Date:	2-21	20-0	No. Units:	12	
Balance Used: AT400			Serial #: 1113	372841				
					·····			
		Storage			Target	Target	Actual	Calc
Compound	CAS	Location	<u>Lot #</u>	Purity	Conc(ug/ml)	<u>Weight</u>	Weight	Conc(ug/ml)
Allyl chloride (	107-05-1	FAIBI3D	00305HO	0.99	2,000.00	200.00	200.00	2,000.00
Chloroprene	126-99-8	FA1D8B	051215JLM	0.99	2,000.00	200.00		0.00
Pentachloroethane	76-01-7	FA1C3B	OGL01	0.98	2,000.00	200.00	200.00	2,000.00
1,1,2-Trichlorotrifluoroetha	76-13-1	FAIAIIA	01404PV	0.99	2,000.00	200.00	200.00	2,000.00
Dichlorodifluoromethane	75-71-8	HOOD	A042007	0.99	2,000.00		4.20 (ml)	1,978.41
Dichlorofluoromethane	75-43-4	HOOD	A042008	0.99	2,000.00		3.10 (ml)	1,974.39
Chlorodifluoromethane	75-45-6	VOA Lab	A042009	0.99	2,000.00		2.40 (ml)	2,016.62
Ethyl acetate	141-78-6	FA1C5B	11073ED	0.99	2,000.00	200.00	200.00	2,000.00
Diisopropyl ether ( DIPE )	108-20-3	FA1C2B	13450CB	0.99	2,000.00	200.00	200.00	2,000.00
Hexachloroethane	67-72-1	RA1B6D	12719A0	0.99	2,000.00	200.00	200.00	2,000.00
Methyl methacrylate	80-62-6	FA1C2D	09505TO	0.99	2,000.00	200.00	200.00	2,000.00
Methacrylonitrile	126-98-7	FA1C2C	04406MI	0.99	2,000.00	200.00	200.00	2,000.00
Diethyl ether (ethyl ether)	60-29-7	FAICIA	17676TQ	0.99	2,000.00	200.00	200.00	2,000.00
2-Nitropropane	79-46-9	RA1C11C	04609PN	0.98	10,000.00	1,000.00	1,000.00	10,000.00
Pr vitrile	107-12-0	FA1C3D	10101EB	0.98	20,000.00	2,000.00	2,000.00	20,000.00
Cycamexanone	108-94-1	RA1D2B	10513PA	0.99	20,000.00	2,000.00	2,000.00	20,000.00
ert-Butanol (TBA)	75-65-0	RA1H2D	06648PC	0.99	40,000.00	4,000.00	4,000.00	40,000.00
l-Butanol	71-36-3	FA1G1B	8238	0.99	80,000.00	8,000.00	8,000.00	80,000.00
sobutanol	78-83-1	FA1C3A	00439HD	0.99	80,000.00	8,000.00	8,000.00	80,000.00
I,4-Dioxane	123-91-1	RA1H3B	03053BD	0.99	80,000.00	8,000.00	8,000.00	80,000.00

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MUSC 74 18->20 Add +

# **CERTIFICATE OF COMPOSITION**

FOR LABORATORY USE ONLY - READ MSDS PRIOR TO USE

1751-77

Lot No.: A042271

Storage: Freezer

110 Benner Circle Bellefonte, PA 16823-8812 Tel: (800) 356-1688 Fax: (814) 353-1309

Description: Custom Volatiles Standard

Catalog No.: 558661

Expiration Date1: July 2007

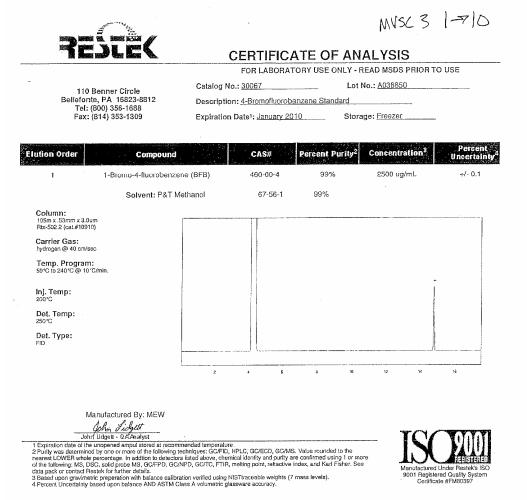
Elution Order	Compound	CAS#	Percent Purity <sup>2</sup>	Concentration <sup>3</sup>	Percent Uncertainty <sup>4</sup>
1	2-Propanol (isopropanol)	67-63-0	99%	20000 ug/mL	+/- 0.1
2	1-Propanol	71-23-8	99%	20000 ug/mL	+/- 0.1
3	n-Hexane (C6)	110-54-3	99%	1000 ug/mL	+/- 0.1
4	Acetaldehyde dimethyl acetal	534-15-6	99%	5000 ug/mL	+/- 0.1
5	Ethyl-tert-butyl ether (ETBE)	637-92-3	99%	1000 ug/mL	+/- 0.1
6	tert-Amyl methyl ether (TAME)	994-05-8	99%	1000 ug/mL	+/- 0.1
7	n-Heptane (C7)	142-82-5	99%	1000 ug/ml.	+/- 0.1
8	2-Chlorobenzotrifluoride	88-16-4	99%	1000 ug/mL	+/- 0.1
9	3-Chlorobenzotrifluoride	98-15-7	99%	1000 ug/mL	+/- 0.1
10	4-Chlorobenzotrifluoride	98-56-6	98%	1000 ug/mL	+/- 0.1
11	3-Chlorotoluene	108-41-8	99%	1000 ug/mL	+/- 0.1
12	1.2,3-Trimethylbenzene	526-73-8	99%	1000 ug/mL	+/- 0.1
13	Dicyclopentadiene	77-73-6	98%	1000 ug/ml.	+/- 0.1
14	1,3.5-Trichlorobenzene	108-70-3	99%	1000 ug/mL	+/- 0 1
	Solvent: P&T Methanol	67-56-1	99%		
Column: 105m x 32mm x 1 Rtx-502 2 (cat #10					
Carrier Gas: helium @ 2 2 milin	niç				
Temp. Progra 40°C (hold 2 min ) @ 8°C/min (hold	to 240°C				
Inj. Temp: 200°C					
Det. Temp: 250°C					
Det. Type: MSO		a   	۳ ۱)		3   1
	6.00 8.00	10.00 12.00 14	00 ±6.00 <sup>°°</sup> 18	00 20 00 22	00 24.00

Manufactured By: FJT

Manufactured By F-J I John Udget - 0.KAstyst 1 Exciration date of the uncoerned ampulsioned at recommended temperature 2 Purity was determined by one or more of the following techniques (C/FID, HPLC, GC/ECD, GC/MS Value rounded to the nearest LOWER whole percentage In addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following MS DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, melting point, reflective index, and Karl Fisher. See data pack or contact Restek for further dotails 3 Based upon gravimetic properation with balance calibration varified using NISTraceable weights (7 mass levels) 4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.

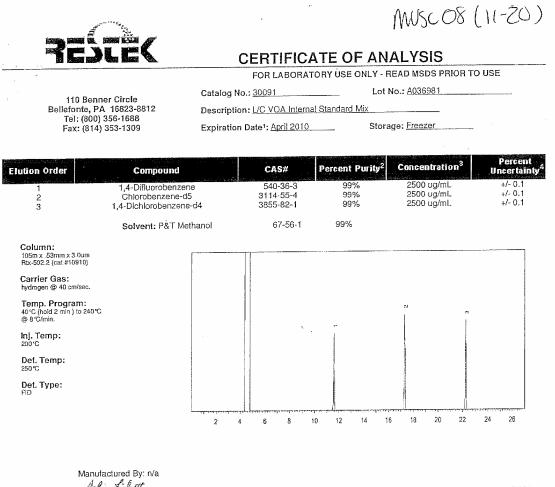


### Table 13. BFB Standard



Page 11

#### Table 14. Internal Standard Mixture

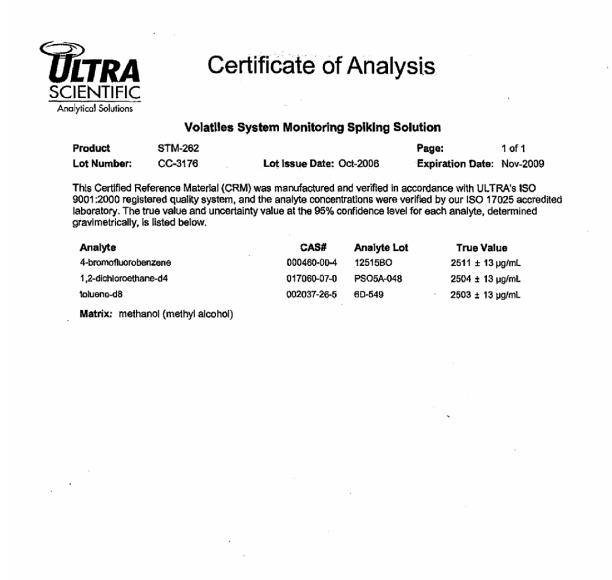


Manufactured By: n/a John Lidget - Q.Konalyst I Expiration date of the unopened ampul stored at recommended temperature. 2 Purity was determined by one or more of the following techniques: GC/FID, HPLC, GC/ECD, GC/MS. Value rounded to the nearest LOWER whole percentage in addition to detectors listed above, chemical identity and purity are confirmed using 1 or more of the following: MS, DSC, solid probe MS, GC/FPD, GC/NPD, GC/TC, FTIR, meting point, refractive index, and Karl Fisher. See data pack or contact Restek for further details. 3 Based upon gravimetric preparation with balance calibration verified using NISTraceable weights (7 mass levels). 4 Percent Uncertainty based upon balance AND ASTM Class A volumetric glassware accuracy.



Page 11

#### Table 15. Surrogate Mixture



Balances used in the manufacture of this standard are calibrated with weights traceable to NIST in compliance with ANSI/NCSL Z-540-1 and ISO 9001.



ISO 17025:2005 Accredited A2LA Cert, No. 0851.01 ISO 9001:2000 Registered TUV USA, Inc. Cart. No. 06-1004 250 Smith Street, North Kingstown, RI 02852 USA 401-294-9400 Fax: 401-295-2330 www.ultrasci.com

See Reverse For Additional Information

William

Quality ssurance planager

**Edison** 



SOP No. ED-MSS-002, Rev. 11 Effective Date: 08/04/2010 Page No.: 1 of 39

# Title: SEMI-VOLATILE ORGANIC COMPOUNDS BY GAS CHROMATOGRAPHY/ MASS SPECTROMETRY (GC/MS), SW846 Method 8270C

Once printed, this is considered an uncontrolled document

Approvals (Signature/Date):						
Wahied Bayoumi SVOA GC/MS Department Manager	08/04/10 Date	Kene' Kasperek Health & Safety Manager	08/04/10 Date			
Caundation Carl Armbruster Quality Assurance Manager	08/04/10 Date	Dru gladwell Ann Gladwell Laboratory Director	08/04/10 Date			

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## 1.0 <u>Scope and Application</u>

## 1.1 Analytes, Matrix(s), and Reporting Limits

USEPA Method 8270C is an analytical method which employs the use of GC/MS to determine the concentration of semivolatile organic compounds in extracts prepared from many types of solid waste matrices, soils, and water samples. TestAmerica Edison has the capability to analyze and report the compounds listed in Table 1 via Method 8270C.

Table 1					
Compound	CAS No.	Compound	CAS No.		
Acenaphthene	83-32-9	4-Chloroaniline	106-47-8		
Acenaphthene-d <sub>10</sub> (IS)		2-Chloronaphthalene	91-58-7		
Acenaphthylene	208-96-8	2-Chlorophenol	95-57-8		
Acetophenone	98-86-2	4-Chlorophenyl phenyl ether	7005-72-3		
Aniline	62-53-3	Chrysene	218-01-9		
Anthracene	120-12-7	Chrysene-d <sub>12</sub> (IS)			
Benzidine	92-87-5	Dibenz(a,h)anthracene	53-70-3		
Benzoic acid	65-85-0	Dibenzofuran	132-64-9		
Benz(a)anthracene	56-55-3	Benzo(g,h,I)perylene	191-24-2		
Benzo(b)fluoranthene	205-99-2	Benzo(a)pyrene	50-32-8		
Benzo(k)fluoranthene	207-08-9	Phenol d6 (surr)			
Benzyl alcohol	100-51-6	Di-n-butyl phthalate	84-74-2		
Bis(2-chloroethoxy)methane	111-91-1	1,2-Dichlorobenzene	95-50-1		
Bis(2-chloroethyl) ether	111-44-4	1,3-Dichlorobenzene	541-73-1		
Bis(2-chloroisopropyl) ether	108-60-1	1,4-Dichlorobenzene	106-46-7		
Bis(2-ethylhexyl) phthalate	117-81-7	1,4-Dichlorobenzene-d <sub>4</sub> (IS)			
4-Bromophenyl phenyl ether	101-55-3	3,3'-Dichlorobenzidine	91-94-1		
Butyl benzyl phthalate	85-68-7	2,4-Dichlorophenol	120-83-2		
2,4-Dimethylphenol	105-67-9	Diethyl phthalate	84-66-2		
Dimethyl phthalate	131-11-3	Hydroquinone	123-31-9		
4,6-Dinitro-2-methylphenol	534-52-1	Indeno(1,2,3-cd)pyrene	193-39-5		
2,4-Dinitrophenol	51-28-5	Isophorone	78-59-1		
2,4-Dinitrotoluene	121-14-2	2-Methylnaphthalene	91-57-6		
2,6-Dinitrotoluene	606-20-2	2-Methylphenol	95-48-7		
1,2-Diphenylhydrazine	122-66-7	3-Methylphenol, 4-Chloro-	59-50-7		
Di-n-octyl phthalate	117-84-0	4-Methylphenol	106-44-5		
Fluoranthene	206-44-0	Naphthalene	91-20-3		
Fluorene	86-73-7	Naphthalene-d <sub>8</sub> (IS)			
2-Fluorobiphenyl (surr)	321-60-8	2-Nitroaniline	88-74-4		
2-Fluorophenol (surr)	367-12-4	3-Nitroaniline	99-09-2		
Hexachlorobenzene	118-74-1	4-Nitroaniline	100-01-6		
Hexachlorobutadiene	87-68-3	Nitrobenzene	98-95-3		
Hexachlorocyclopentadiene	77-47-4	Nitrobenzene-d5 (surr)			
Hexachloroethane	67-72-1	2-Nitrophenol	88-75-5		
N-Nitrosodimethylamine	62-75-9	4-Nitrophenol	100-02-7		
N-Nitrosodiphenylamine	86-30-6	Pyrene	129-00-0		
N-Nitrosodi-n-propylamine	621-64-7	Pyridine	110-86-1		

Table 1				
Compound	CAS No.	Compound	CAS No.	
		o-Toluidine	95-53-4	
Pentachlorophenol	87-86-5	Terphenyl-d <sub>14</sub> (Surr)	1718-51-0	
Perylene-d <sub>12</sub> (IS)		2,4,6-Tribromophenol (surr)	118-79-6	
Phenanthrene	85-01-8	1,2,4-Trichlorobenzene	120-82-1	
Phenanthrene-d <sub>10</sub> (IS)		2,4,5-Trichlorophenol	95-95-4	
Carbazole	86-74-8	2,4,6-Trichlorophenol	88-06-2	
2,4-Dichlorophenol	120-83-2	Phenol	108-95-2	
Atrazine	1912-24-9	Benzaldehyde	100-52-7	
Biphenyl(1,1')	92-52-4	Caprolactam	105-60-2	
1-Naphthylamine	134-32-7	2-Naphthylamine	91-59-8	
n-decane	124-18-5	Coumarin	91-64-5	
2-tert-butyl-4-Methylphenol	2409-55-4	n-Octadecane	593-45-3	
3,5-Di-tert-butyl-4-Hydroxytol	128-37-0	1-Methylnaphthalene	90-12-0	
1,3-Dimethylnaphthalene	575-41-7	Carbamazepine	298-46-4	
N,N-Dimethylaniline	121-69-7	Diphenyl Ether	101-84-8	
1,4-Dioxane	123-91-1	1,2,4,5-Tetrachlorobenzene	95-94-3	
2,3,4,6-Tetrachlorophenol	58-90-2			

- **1.2** For a listing of method detection limits (MDLs) and Reporting Limits (RLs) please refer to the currently active Method 8270C Method Limit Group in TALS (TestAmerica LIMS).
- **1.3** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Section 7 (*Review of Work*), and Section 20 (*Test Methods and Method Validation*) in TestAmerica Edison's Quality Assurance Manual (TestAmerica Edison Document No. ED-QA-LQM).

## 2.0 <u>Summary of Method</u>

- **2.1** This method is used for the analysis of aqueous and solid matrices for semivolatile base, neutral and acid organic compounds that are extracted from the sample matrix with an organic solvent.
- **2.2** An aliquot of sample containing surrogate spiking compounds is extracted with an organic solvent. The extract is concentrated on a steam bath to a suitable volume. Internal standards are added to the extract.
- **2.3** Sample extraction techniques are specified for each matrix in the following TestAmerica Edison SOPs:
  - ED-ORP-002 (Extraction of Semivolatile Organic Compounds in Water by Separatory Funnel, SW846 Method 3510C);
  - ED-ORP-004 (Automated Soxhlet Extraction of Solid Samples Semivolatile Compounds, SW846 Method 3541);
  - ED-ORP-005 (Extraction of Semivolatile Compounds in Soil Using Low-Level Extraction Techniques, SW846 Method 3550B);
  - ED-ORP-006 (Extraction of Semivolatile Compounds in Soil Using Medium Level Extraction Techniques, SW846 Method 3550B).

- **2.4** A small aliquot of the extract is injected into a gas chromatograph (GC) equipped with a capillary column. The GC is temperature programmed to separate the compounds by boiling point, which were recovered during the extraction step. The effluent of the gas chromatograph is interfaced to a mass spectrometer (MS) which is used to detect the compounds eluting from the GC. The detected compounds are fragmented with an electron beam to produce a mass spectrum which is characteristic of the compound introduced into the MS. Identification of target analytes is accomplished by comparing their mass spectra with the electron ionization spectra of authentic standards. Quantitation is accomplished by comparing the response of a major ion (quantitation ion) relative to an internal standard established through a five-point calibration (six points for second order regression). Specific calibration and quality control steps are included in the method that must be performed and must meet the specifications of SW846 Method 8270C.
- **2.5** This method is also applicable to the analysis of samples by Selected Ion Monitoring (SIM) for the purpose of obtaining lower reporting limits for the following compounds: Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(k)fluoranthene, Dibenz(a,h)anthracene, Hexachlorobenzene, Indeno(1,2,3-c,d)pyrene, Pentachlorophenol and N-Nitroso-di-methylamine.

## 3.0 <u>Definitions</u>

For a complete list of definitions refer to Appendix 2 in the most current revision of the Quality Assurance Manual (ED-QA-LQM).

## 4.0 Interferences

- **4.1** GC/MS data from all blanks, samples, and spikes must be evaluated for interferences. Analysts must take steps to determine the source of the interference and take corrective action to eliminate the problem.
  - **4.1.1** Contamination by carryover can occur whenever highconcentration and low-concentration samples are sequentially analyzed. To reduce carryover, the sample syringe is automatically rinsed with solvent between sample injections. Whenever an unusually concentrated sample is encountered, it should be followed by the analysis of a solvent blank to check for cross-contamination. Alternately, verify that the sample analyzed after the high concentration sample does not show any carryover through inspection of chromatogram and target results.
  - **4.1.2** Contaminants from the extraction process, detected in the method blank should be evaluated to determine the impact on the analysis. Interferences from any target analyte must not be present in the method blank above the reporting limit for that compound. If these types of interferences occur, corrective action is required. The source should be identified and

corrective action initiated to eliminate the interference from the extraction process. Affected samples must be re-extracted and re-analyzed.

- **4.1.3** The analyst must take precautions to make sure that contaminants do not enter the analytical system. These precautions include systematic procedures designed to eliminate interferences.
- **4.2** Some compounds analyzed using this method are unstable or sensitive. Benzidine, for example, is easily oxidized during extraction. Hexachlorocyclopentadiene breaks down photochemically and can decompose from high temperatures, particularly in the injection port of the GC. Phenols are sensitive to active sites and can give a low response or exhibit poor chromatography by tailing. Therefore, it is important the GC is maintained in the best possible condition. See Section 9.2.2 for proper daily maintenance.

## 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

## 5.1. Specific Safety Concerns or Requirements

The gas chromatograph and mass spectrometer contain zones that have elevated temperatures. The analyst needs to be aware of the locations of those zones, and must cool them to room temperature prior to working on them.

The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

There are areas of high voltage in both the gas chromatograph and the mass spectrometer. Depending on the type of work involved, either turn the power to the instrument off, or disconnect it from its source of power.

## 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure		
Methanol	Flammable Poison Irritant	200 ppm- TWA	A slight irritant to the mucous membranes. Toxic effects exerted upon nervous system, particularly the optic nerve. Symptoms of overexposure may include headache, drowsiness and dizziness. Methyl alcohol is a defatting agent and may cause skin to become dry and cracked. Skin absorption can occur; symptoms may parallel inhalation exposure. Irritant to the eyes.		
Methylene Chloride	Carcinogen Irritant	25 ppm- TWA 125 ppm- STEL	Causes irritation to respiratory tract. Has a strong narcotic effect with symptoms of mental confusion, light-headedness, fatigue, nausea, vomiting and headache. Causes irritation, redness and pain to the skin and eyes. Prolonged contact can cause burns. Liquid degreases the skin. May be absorbed through skin.		
Toluene	Flammable Poison Irritant	200 ppm- TWA 300 ppm- Ceiling	Inhalation may cause irritation of the upper respiratory tract. Symptoms of overexposure may include fatigue, confusion, headache, dizziness and drowsiness. Peculiar skin sensations (e. g. pins and needles) or numbness may be produced. Causes severe eye and skin irritation with redness and pain. May be absorbed through the skin.		
Dimethyl- dichloro- silane	Flammable	none	Can be corrosive to the respiratory tract causing severe irritation and tissue damage. Harmful if absorbed through the skin. May cause severe irritation and systemic damage. Severely irritating to the skin and eyes. Harmful if swallowed. Can cause abdominal discomfort, nausea, vomiting, diarrhea, and irritation to the mouth, throat and stomach.		
<ol> <li>Always add acid to water to prevent violent reactions.</li> <li>Exposure limit refers to the OSHA regulatory exposure limit.</li> </ol>					
2 – Exposure lim	it refers to the	OSHA regula	tory exposure limit.		

# 6.0 Equipment and Supplies

- 6.1 Gas chromatograph/mass spectrometer system
  - **6.1.1** Gas chromatograph: An Agilent/HP 5890 (or equivalent) houses the capillary column. The GC provides a splitless injection port and allows the column to be directly coupled to the mass spectrometer. The oven is temperature programmable to meet the requirements of the method. An HP 7673 autosampler (or equivalent) with a 10 ul syringe provides automatic injection of sample extracts while the instrument is unattended.

- **6.1.2** Analytical Column: 30m x 0.25mm ID, 0.25 um film thickness, Restek Rxi-5Sil MS, Catalog #13623.
- Mass spectrometer: Agilent (HP) 5972, 5973 or 5975 Mass 6.1.3 Selective Detector (MSD) Capable of scanning from 35 to 500 amu every 1 sec or less, using 70 volts electron energy in the electron ionization mode. The mass spectrometer must be capable of producing mass spectrum for 50 of а na decafluorotriphenylphosphine (DFTPP) which meets the criteria in Section 9.2.3 when 2 ul of the 25 ug/ml GC/MS tuning standard is injected through the GC.
- **6.1.4** GC/MS interface: Any GC-to-MS interface may be used that gives acceptable calibration points at 50 ng per injection for each compound of interest and achieves acceptable tuning performance criteria.
- 6.1.5 Data system: The data system is interfaced to the mass spectrometer and accommodates continuous acquisition and storage of GC/MS data throughout the duration of the chromatographic program. The data system consists of a Hewlett-Packard Chemstation equipped with Mustang software used for instrument control and data acquisition. This, in turn, is interfaced to Thruput's Unix hardware equipped with Target software for data processing. Data from sample extract analysis can be accessed in real-time, while sample data reports and library searches can be performed on data files from previously run samples. The software is also capable of searching any GC/MS data file for ions of a specific mass whose abundances can be plotted versus time or scan number which allows integration of abundances for any extracted ion between specified times or scan-number limits. Librarv searches utilize a NIST 02.1 Mass Spectral Library.
- **6.2** Bottles, glass with polytetrafluoroethylene (PTFE)-lined screw caps or crimp tops.
- **6.3** Injection port liners, splitless
- 6.4 Injection port septa
- 6.5 Injection port graphite seals
- **6.6** Pre-silanized glass wool (Supelco 2-0411 or equivalent)
- 6.7 Syringes, Assorted sizes 10ul 1000ul; gas-tight
- 6.8 Bottles, 10 and 5ml amber screw cap with Teflon liner
- 6.9 Vials, 2ml amber screw cap with Teflon liner

- **6.10** Wheaton microvials 100ul (or equivalent)
- **6.11** Volumetric Flasks, Class A with ground glass stoppers (2ml 50ml)
- **6.12** Analytical balance, ASP Model SP-180 (or equivalent), capable of accurately weighing to 0.0001 gr.

## 7.0 <u>Reagents and Standards</u>

### 7.1. Reagents:

- **7.1.1.** Methylene Chloride: J.T.Baker Resi-Analyzed, used for Organic Residue Analysis (P/N 9266-V8 or equivalent).
- 7.1.2. Methanol: J.T.Baker Purge and Trap Grade (P/N 9077-02 or equivalent).
- **7.1.3.** Toluene: J.T.Baker Resi-Analyzed, for Organic Residue Analysis (P/N 9460-03 or equivalent).
- **7.1.4.** Sylon-CT: Supelco (P/N 33065-U or equivalent). Sylon-CT is a highly reactive silanizing reagent consisting of 95% Toluene and 5% Dimethyldichlorosilane (DMDCS).
- **7.1.5.** Each lot of solvent is screened for contaminants before being used for analysis as detailed in TestAmerica Corporate Quality SOP No. CA-Q-S-001 (*Solvent & Acid Lot Testing & Approval*) and TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*).

### 7.2. Standards:

**7.2.1. Calibration Standards (full scan)**: Stock analytical standard solutions are purchased mainly from Supelco Analytical. Other standards are prepared in the laboratory as needed using neat compounds or prepared solutions purchased from SPEX CertiPrep, Chem Service, Accustandard, Supelco or other suppliers. Secondary dilutions are either made from purchased Supelco Analytical stock solutions are listed below or from prepared solutions as listed in the following table:

(NOTE: Second sources (from separate lots) are used for quanitation standards and spiking/ICV standards).

Target Analyte Standard Name	Concentration	Vendor	Catalog #
Custom Acid Mix 1	2000ppm	Supelco	86-1213
Custom Acid Mix 1 (Second source)	2000pmm	Supelco	86-1218
Custom Acid Mix 2	2000ppm	Supelco	86-1214

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Target Analyte Standard Name	Concentration	Vendor	Catalog #
Custom Acid Mix 2 (Second source)	2000ppm	Supelco	86-1220
Custom Base Neutral Mix 1	(VARIED)	Supelco	86-1212
Custom Base Neutral Mix 2	2000ppm	Supelco	86-1216
Custom Base Neutral Mix 2 (Second source)	2000ppm	Supelco	86-1217
Benzidine	5000ppm	Supelco	4-0005
Benzidine (Second source)	5000ppm	Supelco	86-1221
3,3'-Dichlorobenzidine	5000ppm	Supelco	4-0026
3,3'-Dichlorobenzidine (Second source)	5000ppm	Supelco	86-1222
N-Nitrosodiphenylamine	5000ppm	Supelco	46702-U
N-Nitrosodiphenylamine (Second source)	5000ppm	Supelco	86-1227
OLM04 Semivolatile Mix	2000ppm	Supelco	47514-U
OLM04 Semivolatile Mix (Second source)	2000ppm	Supelco	86-1231
1,2,3,4-TCDD	50ppm	SPEX	DD1234-50
SPEX Special Mix (contains compounds	2000ppm	SPEX	XQ-4616
listed below)			
SPEX Custom Mix (contains compounds	2000ppm	SPEX	XQ-4555
listed below)			
1,2,4,5-Tetrachlorobenzene	1000ppm	Absolute	70274
2,3,4,6-Tetrachlorophenol	1000ppm	Absolute	70477

Spex Special Mix
o-Toluidine
1-Naphthylamine
2-Naphthylamine

TestAmerica Custom Mix
n-decane
2-tert-butyl-4-Methylphenol
3,5-Di-tert-butyl-4-Hydroxytol
Coumarin
n-octadecane
1-Methylnaphthalene
N-Methylaniline
Carbamazepine
Benzonitrile
1,3-DimethyInaphthalene

**7.2.1.1.** Prepare 10ml working solutions at concentrations indicated in Table 2, Working Standards Preparation (see Tables section in rear of document) Prepare by combining the indicated volumes of each stock solution into a 10 ml volumetric flask. Dilute to the volume marker with Methylene Chloride.

- **7.2.1.2.** Initial Calibration Verification (full scan): Prepare a second source ICV working solution as described in Table 2. The second source 25ppm ICV (full scan) is prepared by adding 6.25ml of the 100 ppm working solution to a 25 ml volumetric flask and bringing it to volume with Methylene Chloride.
- **7.2.2.** Calibration Standards (SIM analysis): The Edison lab currently analyzes only a select list of compounds by 8270 SIM.
  - **7.2.2.1** Initial Calibration Standards: Prepare SIM working solutions as indicated in Table 3, SIM Working Standards Preparation.

Standard Name	Concentration	Vendor	Catalog #
Pentachlorophenol	100ppm	Accustandard	App-9-176
n-Nitrosodimethylamine	100ppm	Accustandard	APP-9-149
Hexachlorobenzene	100ppm*	Accustandard	APP-9-112
PAH Mix	100ppm	Accustandard	M-610

\*Hexachlorobenzene is diluted to 10ppm prior to SIM Standards prep

**7.2.2.2** SIM Initial Calibration Verification (ICV): The 0.1 ppm separate source SIM ICV is prepared as detailed in Table 6 using the following standards purchased from ChemService (or equivalent):

Standard Name	Concentration	Vendor	Catalog #
Pentachlorophenol	100ppm	ChemService	F64JS
n-Nitrosodimethylamine	100ppm	ChemService	F61JS
Hexachlorobenzene	100ppm	ChemService	F9JS
PAH Mix	100ppm	ChemService	PPH-10JM

- **7.2.2.3** Internal Standard solution for SIM analysis is prepared at 50 ppm by adding 125ul of the 2000ppm stock ISTD (see Section 7.2.3) and bringing to volume with Methylene Chloride in a 5ml volumetric flask.
  - **7.2.2.3.1** For SIM analysis inject 20ul of this solution (50ppm) per ml of sample extract prior to analysis resulting in a concentration of 1ppm (ug/ml) in the extract.
- **7.2.3. Surrogate Standards**: A 2000ppm Acid Surrogate Standard and a 2000ppm BN Surrogate Standard is purchased from Supelco Analytical for use in spiking blanks, samples and associated QC prior to extraction.

Standard Name	Vendor	Catalog #
Base Neutral Surrogate	Supelco	86-1252
Acid Surrogate	Supelco	86-1249

**7.2.4.** Internal Standards (full scan analysis): The Internal Standards Solution at 2000ppm is purchased from Supelco (Catalog # 86-1238).

Internal Standard Compounds				
1,4-Dichlorobenzene-d4				
Phenanthrene-d10				
Naphthalene-d8				
Chrysene-d12				
Acenaphthene-d10				
Perylene-d12				

- **7.2.4.1.** The Internal Standard solution is stored in 10ml amber screw cap bottles with Teflon liners in the dark at 4°C. The Internal standard solution is used in preparing all analytical standards. Inject 20ul of this solution (2000ppm) per ml of sample extract prior to analysis resulting in a concentration of 40ppm (ug/ml) in the extract.
- **7.2.5. GC/MS Instrument Performance Check (DFTPP):** The DFTPP standard is prepared by is prepared at 25 ppm by adding 2.5ml of EPA 8270 GC/MS Tuning Solution II (Supelco Catalog # 47548-U) to a 100ml volumetric flask and bringing to volume with Methylene Chloride.
- **7.2.6.** Information on prepared standard solutions must be recorded in a standards logbook or in the TALS Reagent Module. Information such as standard supplier, lot number, original concentration, a description of how the standard was made, are required along with the laboratory lot number, analyst's initials, date prepared, expiration date and verification signature. Standards must be remade every 6 months, or sooner, if the standards expire or begin to show signs of unacceptable degradation. Class "A" volumetric must be used at all times and syringes, preferably gas-tight syringes when available, should be checked for accuracy using an analytical balance. Class "A" pipettes should also be used if volumes permit.

# 8.0 <u>Sample Collection, Preservation, Shipment and Storage</u>

- 8.1 All samples must be stored at  $4^{\circ}C (\pm 2^{\circ}C)$  upon receipt.
- **8.2** Sample Extract Storage. Samples extracts must be protected from light and refrigerated at  $4^{\circ}C$  ( $\pm 2^{\circ}C$ ) from time of extraction until analysis.
- **8.3** Sample Extract Holding Time. All sample extracts must be analyzed within 40 days of extraction.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time	Reference
Waters	Amber glass, 1L	1000 ml	Cool 4 <u>+</u> 2ºC	7 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C
Solids	Wide mouth glass, 8 or 16 oz.	50g	Cool 4 <u>+</u> 2ºC	14 days to extraction; Analyze within 40 days of extraction	EPA Method SW846 8270C

### 9.0 <u>Quality Control</u>

**9.1.** <u>Sample QC</u> - The following quality control samples are prepared with each batch of samples.

Quality Controls	Frequency	Control Limit
Method Blank (MB)	1 in 20 or fewer samples	< Rpt. Limit
Laboratory Control Sample (LCS) <sup>1</sup>	1 in 20 or fewer samples	Statistical Limits <sup>4</sup>
Matrix Spike (MS) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits <sup>4</sup>
MS Duplicate (MSD) <sup>2</sup>	1 in 20 or fewer samples	Statistical Limits <sup>4</sup>
Surrogates	every sample <sup>3</sup>	Statistical Limits <sup>4</sup>
Internal Standards	Every sample	Response within -50% to +100% of CCV

<sup>1</sup> LCS Duplicate (LCD) is performed only when insufficient sample is available for the MS/MSD or when requested by the client/project/contract.

<sup>2</sup> The sample selection for MS/MSD are randomly selected, unless specifically requested by a client....predetermined by the extraction lab.

<sup>3</sup> Analytical and QC samples (MB, LCS, MS/MSD, Method Blank)

<sup>4</sup> Statistical control limits are updated annually and are updated into lab reporting software.

**9.1.1. Method blanks** are extracted with every sample batch on each day that samples are extracted. To be considered acceptable, the method blank must contain less than the reporting limit of all target compounds.

If method blanks are unacceptably contaminated with target compounds that are also present in field samples, all affected samples must be reextracted and re-analyzed. Corrective action must be taken to identify and eliminate the contamination source. Demonstrate that acceptable blanks can be obtained before continuing with sample extraction and analysis. Method blanks must be analyzed on each instrument on which the associated samples are analyzed.

- **9.1.1.1.** Surrogate recoveries for the method blank must be within the laboratory generated limits. If any surrogate is outside the limits, the method blank must re-analyzed. If any surrogate is still outside limits, all samples and QC samples associated with that method blank must be re-extracted (volume permitting).
- **9.1.2. Matrix Spike (MS)/Matrix Spike Duplicate (MSD):** A matrix spike/matrix spike duplicate (MS/MSD) pair is extracted and analyzed with every 20 environmental samples of a specific matrix (defined as a sample batch). Full compound list spiking is employed for MS/MSDs and LCSs. These spikes are prepared and extracted concurrent with sample preparation. MS and MSD recoveries are calculated and compared to lab generated acceptance criteria. See the current active TALS 8270C Method Limit Group for QC limits. A minimum of 16 spiked analytes are reported to in client reports (the full list is reported at least once during each 2 year period).
  - **9.1.2.1** Spike recovery limits are lab generated and are updated annually.
  - **9.1.2.2** An LCS/LCSD may be substituted for the MS/MSD if insufficient sample volume is available.
- **9.1.3.** Laboratory Control Sample (LCS)/Laboratory Control Sample Duplicate (LCSD): A Laboratory Control Sample (LCS) (aka blank spike) must be extracted and analyzed with each batch of 20 environmental samples. The LCS data is used to assess method performance if the MS/MSD recoveries fall outside of the lab generated limits (See the current active TALS 8270C Method Limit Group for QC limits). If the LCS recovery is within the current lab generated limits, the MS/MSD recoveries are attributed to matrix interference. If the LCS recovery results are outside the method specified, the LCS extract is reanalyzed. If, upon reanalysis, the LCS is it is still outside of limits the entire batch must be re-extracted and reanalyzed.
  - **9.1.3.1** A Laboratory Control Sample Duplicate (LCSD) is extracted and analyzed only when insufficient client sample is available for preparation of an MS/MSD pair. The LCS/LSCD is evaluated in the same manner as the MS/MSD (see Section 9.1.2)
- **9.1.4.** Surrogate Standards: All samples, blanks and QC samples are spiked with a six (6) component surrogate standard mix (see Section 7.2.3). The percent recovery of the surrogate standards is calculated and compared to lab generated limits (See the current active TALS 8270C Method Limit Group for QC limits).

If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, the sample must be re-extracted and re-analyzed to confirm matrix

interference. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary.

- **9.1.4.1** Surrogate recovery limits are lab generated and are updated annually
- **9.1.5. Internal Standards**: The response (area count) of each internal standard in the sample must be within -50 +100% of its corresponding internal standard in the CCV or, the ICAL midpoint for samples analyzed under the initial calibration range. Failure to meet these criteria is indicative of sample matrix effects. All samples failing these criteria must be reanalyzed to confirm matrix effects.

## 9.2. Instrument QC

**9.2.1 GC/MS Instrument Performance Check (DFTPP)**: The GC/MS system is tuned using Perfluortributylamine (PFTBA) such that an injection of 50ng of Decafluorotriphenylphosphine (DFTPP) meet the abundance criteria listed in the table below. Prior to the analysis of any calibration standards or samples, the GC/MS system must meet all DFTPP key ion abundance criteria. This analysis will verify proper tuning of the system for a period of 12 hours post-injection. After 12 hours, the instrument performance must again be verified prior to the analysis of standards, QC or samples.

DFTPP Key lons and Abundance Criteria		
Mass	Ion Abundance Criteria	
51	30-60% of mass 198	
68	<2% of mass 69	
69	reference only	
70	<2% of moss 69	
127	40-60% of mass 198	
197	<1% of mass 198	
198	Base Peak, 100% relative abundance	
199	5-9% of mass 198	
275	10-30% of mass 198	
365	>1% of mass 198	
411	0-100% of mass 443	
442	>40% of mass 198	
433	17-23% of mass 442	

- **9.2.1.1.** Evaluate DFTPP using three scan averaging and background subtraction techniques. Select the scan at the peak apex, add +1 scan from the apex and -1 scans from the apex.
- **9.2.1.2.** The mass spectrum of DFTPP may be background subtracted to eliminate column bleed or instrument background ions. Background subtract DFTPP by selecting a scan for subtraction ≤20 scans <u>before</u> the apex scan of DFTPP.

- **9.2.1.3.** Check column performance using pentachlorophenol and the benzidine peaks (these compounds are included in the DFTPP solution). Benzidine & Pentachlorophenol should respond normally without significant peak tailing (<3). If responses are poor and excessive peak tailing is present, corrective action for the GC/MS instrument may be required. Corrective actions may include:
  - 9.2.1.3.1 Retune the GC/MS;
    9.2.1.3.2 Clip the injector end of the GC column;
    9.2.1.3.3 Replace the septum and injection port liner;
    9.2.1.3.4 Change the injection port seal;
    9.2.1.3.5 Replace the GC column;
    9.2.1.3.6 Clean the injection port with MeCl2
    9.2.1.3.7 Clean the MS ion source;
    9.2.1.3.8 Place a service call.
- **9.2.1.4.** The breakdown of 4, 4-DDT into 4,4-DDD and 4,4'DDE may also be used to assess GC column performance and injection port inertness. If so evaluated the breakdown must be <20%.
- **9.2.1.5.** DFTPP parameter settings are stored in a tune file, which will be used in all subsequent analysis of standards and sample extracts.

# 9.2.2 Initial Calibration Range and Initial Calibration Verification

- **9.2.2.1. Initial Calibration:** The initial calibration range consists of a five-point concentration (six points for second order regression) range of analytical standards prepared as described in Section 7.2.1 and Table 2 and analyzed once the DFTPP instrument performance check has met the criteria in Section 9.2.1.
- **9.2.2.2.** Initial Calibration Verification (ICV): An Initial Calibration Verification (ICV) standard is analyzed immediately after the Initial Calibration Range and before any samples are analyzed. The ICV is prepared as detailed in Section 7.2.2 and Table 3. The ICV must be from a source (or lot) separate from the standards used in the Initial Calibration Range.
- **9.2.3** Continuing Calibration Verification (CCV): A mid-point (appx. 50 ug/ml) Continuing Calibration Verification (CCV) must be analyzed every 12 hours after the DFTPP instrument performance check. The CCV is prepared as detailed in Section 7.2 and Table 3.

## 9.2.4 Calibration Acceptance Summary

**9.2.4.1. Retention Time Windows:** Retention time windows must be established to compensate for minor shifts in absolute retention times as a result of sample loading and normal chromatographic variability. Obtain the retention time for all compounds from the

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analysis of the midpoint standard for the calibration curve. Establish the center of the retention time window by using the absolute retention time for each analyte, internal standard and surrogate from the calibration verification standard at the beginning of the analytical shift. For samples run during the same shift as an initial calibration, use the retention time of the mid-point standard of the initial calibration. For qualitative identification to be acceptable the retention time of the relative retention time (automatically calculated in Target) must be within 0.8 - 1.2 RRT units of its assigned internal standard. The relative retention times of each compound in the five calibration standards must agree within .06 relative retention time units.

**9.2.4.2. Initial Calibration Range:** Internal standard calibration is employed for this method. After the initial calibration range has been analyzed the relative response factor (RRF) for each target/surrogate compound at each concentration level is determined using the following equation.

$$RRF = \underline{A_x} \times \underline{C_{is}} \\ \overline{A_{is}} \quad \overline{C_x}$$

Where:

- $A_x$  = Area characteristic ion for the compound (see Table 1)
- Ais = Area characteristic ion of associated internal standard (See Table 2)
- Cis = Concentration of internal standard
- Cx = Concentration of compound in standard
  - 9.2.4.2.1. Determine the mean RRF for each compound using the five RFs from the initial calibration range. The average RFs of the four (4) System Performance Check Compounds (SPCCs) listed in Table 4 must be ≥ 0.050.
  - **9.2.4.2.2.** Calculate the Standard Deviation (SD) and Percent Relative Standard Deviation (% RSD) of the response factors for each compound:

% RSD = <u>Standard Deviation of RRFs</u> Mean RRF

9.2.4.2.3. The % RSD of the RRF's must be <15% for each target analyte. The % RSD of the thirteen (13) Calibration Check Compounds (CCCs) listed in Table 5 must be ≤30% in order for the calibration range to be acceptable. If the % RSD of any of the CCCs is ≥30% the calibration has failed and corrective action must be performed.</p>

- **9.2.4.2.4.** If the above listed criteria is met, the system can be assumed to be linear and sample analysis may begin and the average RF from the initial calibration range is used to quantitate all samples.
- **9.2.4.2.5.** An alternative calibration technique may be employed for those any compounds exceeding the 15% RSD criteria:
  - 9.2.4.2.5.1 Calculate the first order linear regression for any compound which did not meet the 15% criteria. First order linear regression calibration may be employed if alternative average response calibration procedures were not applicable. The r value (Correlation Coefficient) of the equation must be ≥0.99 for the calibration to be employed.
  - **9.2.4.2.5.2** Second order regression calibration can be used for any compound that has an established history as a non-linear performer.
  - **9.2.4.2.5.3** If second order regression calibration is used an additional sixth (6th) calibration standard must be analyzed.
  - **9.2.4.2.5.4** If second order regression calibration is used, the  $r^2$  (Correlation Coefficient) value must be  $\geq 0.99$
- **9.2.4.3.** Initial Calibration Verification (ICV):.Once the initial calibration has been analyzed and has met the above criteria, a second source Initial Calibration Verification (ICV) (as prepared in Section 7.2) must be analyzed and evaluated. The ICV must meet the criteria of 80-120% recovery for all compounds however up to 20% of the compounds are allowed exceed this criteria as long as their recoveries are within 65-135%. If corrective action is required check the standard solution, perform instrument maintenance, etc. and re-inject the ICV. If the %D still exceeds 20% after a single ICV reinjection, a new Initial Calibration Range must be analyzed (again, 20% of the compounds are allowed to exceed 20% as long as recoveries are within 65-135%).
- **9.2.4.4.** Continuing Calibration Verification (CCV): A CCV consisting of a standard at or near the midpoint of the Initial Calibration Range is analyzed every 12 hours of instrument operation or at the beginning of an analytical sequence to verify the initial calibration. The calibration verification consists of a DFTPP instrument performance check, and analysis of a calibration verification standard.

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- **9.2.4.4.1** Tune Verification: Follow the procedure for verifying the instrument tune described in section 9.2.3 using a 50 ng injection of DFTPP. If the tune cannot be verified, analysis must be stopped, corrective action taken and a return to "control" demonstrated before continuing with the calibration verification process.
- **9.2.4.4.2** Calibration Verification: Analyze the calibration verification standard immediately after a DFTPP that meets criteria. Use the mid point calibration standard (approximately 50ug/l). <u>NOTE</u>: The calibration standard contains internal standards; Dichlorobenzene  $d_4$ , Naphthalene  $d_8$ , Acenaphthene  $d_{10}$ , Phenanthrene  $d_{10}$ , Chrysene  $d_{12}$ , and Perylene  $d_{12}$  at 40ug/l (0.1ug/L for SIM). The calibration check standard must also include all the target analytes from the original calibration.
- **9.2.4.4.3** The RFs of the four (4) System Performance Check Compounds (SPCCs, See Table 4) must  $be \ge .050$ .
- **9.2.4.4.4** The percent difference (when using average response factor) or percent drift (when using linear regression) of the thirteen (13) Calibration Check Compounds (CCCs, see Table 5) must be  $\leq 20\%$ .
- **9.2.4.4.5** If CCCs were not among the project analytes, all target analytes must meet the 20% D or 20% drift criteria.
- **9.2.4.4.6** The retention times of the internal standards from the calibration check must be within ±30 seconds of the internal standards from the mid point standard of the original calibration. If the retention time for any internal standard changes by more than 30 seconds from the latest daily (12 hour) calibration standard, the chromatographic system is inspected for malfunctions, and corrections made as required. If corrective action does not result in the retention time criteria being achieved, the system must be re-calibrated using four additional standards.
- **9.2.4.4.6** The response (area count) of each internal standard in the calibration verification standard must be within 50 100% of its corresponding internal standard in the mid-level calibration standard of the active calibration curve. If the

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EICP area for any internal standard changes by more than a factor of two (-50% +100%), the mass spectrometer system must be inspected for malfunction and corrections made as appropriate. When corrections are made, re-analysis of samples analyzed while the system was malfunctioning is required.

- **9.2.4.4.7** The relative retention times of each compound in the calibration verification standard must agree within .06 relative retention time units of its value in the initial calibration.
- **9.2.4.4.8** Use the average response factors from the original five-point calibration for quantitative analysis of target analytes identified in field samples.
- **9.2.4.4.8** Prepare a calibration summary or list indicating which compounds did not meet the 20% average percent difference criteria. Record this information in that run log.

### 10.0 Procedure

### 10.1. Gas Chromatograph/Mass Spectrometer Operation

- **10.1.1.** The sequence of events for GC/MS analysis involves many steps. First the injection system and column performance and calibration must be verified. Maintenance operations are performed as needed.
- **10.1.2. Preparation of the Injection Port Liner and Installation Procedure**: Prior to the start of initial calibration and each daily analysis of sample extracts, a new liner for the injection port must be prepared. Once a liner has been used it is no longer inert and will cause serious chromatography problems with phenols and other compounds. When preparing the liner, proper laboratory protection must be worn and the liner must be prepared in a well-ventilated hood. When the procedure is completed all traces of toluene, Sylon-Ct and methanol will be removed immediately so that extraction solvents and preparation of sample extracts will not come into contact with these solvents and become contaminated.
  - **10.1.2.1** Remove one liner from a 40ml VOA bottle containing other liners immersed in Sylon-Ct solution. Rinse off the liner with Toluene and wipe dry. Insert 1cm of pre-silanized glass wool partially into one end of the liner and trim neatly. Push the glass wool into the center of the liner so that it is 1 1/4" from the bottom. Do not use glass wool or solvents that are dirty (i.e. suspended particles) or use liners which are chipped on the

ends, deformed or fractured. Inspect the glass wool for cleanliness after it has been inserted.

- **10.1.2.2** Using a Pasteur pipette flush out the interior of the liner containing the glass wool with Sylon-Ct. Rest the liner horizontally on a small beaker and allow the Sylon-Ct to redeactivate the interior surfaces and the glass wool. There should be no air bubbles caught in the glass wool. After several minutes flush out the Sylon-Ct with toluene and finally with methanol. Dry the outer surface of the liner and rest it on the injection port housing until the remaining methanol is boiled off
- **10.1.2.3** Insert the liner with the newly silanized glass wool plug into the injection port. Verify that the column extends up into the injection port and is perpendicular. Inspect the graphite seal and replace it if the edges are knife-shaped.
- **10.1.2.4** The septum is always replaced daily. Bake out the column at 300°C for 15 minutes after the vacuum in the analyzer has returned to normal.
- **10.1.2.5** Performance may enhanced by clipping a small portion of the column at the injection port end. Document this activity in the maintenance record.
- **10.1.3.** Prior to calibration or sample analysis always verify that the analyzer is under sufficient vacuum and that the column has proper carrier gas flow.
- **10.1.4.** Establish the following GC/MS operating conditions:

Full Scan Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300 <sup>0</sup> C
Source Temperature: Preset by H.P. at 280 <sup>0</sup> C
Scan start time: 1.0 minutes
Initial Column Temperature and Hold Time:
45 <sup>o</sup> C for 0.5 minutes
Column Temperature Program:
20°C /min to 100°C
25°C/min to 270°C
10° C/min to 310°C
Final Column Temperature Hold: 310 <sup>o</sup> C for 5 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 275 <sup>0</sup> C
Injector: Grob-type, splitless

Injection Volume: 1ul
Splitless Valve Time: 0.5 minutes

# **10.1.4.2** SIM Operating Mode

SIM Mode
Mass Range: 35 to 500amu
Scan Time: 1 sec/scan
Transfer Line Temperature: 300 <sup>o</sup> C
Source Temperature: Preset by H.P. at 280 <sup>0</sup> C
Scan start time: 1.5 minutes
Initial Column Temperature and Hold Time:
45 <sup>0</sup> C for 0.5 minutes
Column Temperature Program:
20°C /min to 100°C
25°C/min to 270°C
10° C/min to 310°C
Final Column Temperature Hold: 310 <sup>0</sup> C for 3 minutes
Carrier Gas: Ultra High Purity Grade Helium at 1.3ml/min
Injector Temperature: 300 <sup>o</sup> C
Injector: Grob-type, splitless
Injection Volume: 1ul
Splitless Valve Time: 0.5 minutes

# SIM Parameters

Group 1 Plot 1 Ion: 74.0 Ions/Dwell in Group	(Mass Dwell) 42.0 50 74.0 50 136.0 50	(Mass Dwell) 43. 0 50 128.0 50 150.0 50	(Mass Dwell) 68.0 50 129.0 50 152.0 50
Group 2 Group Start Time: 6.00 Plot 1 Ion: 152.0 Ions/Dwell in Group	(Mass Dwell) 151.0 50 154.0 50 165.0 50	(Mass Dwell) 152.0 50 162.0 50 166.0 50	(Mass Dwell) 153.0 50 164.0 50
Group 3 Group Start Time: 7.80 Plot 1 Ion: 188.0 Ions/Dwell in Group	(Mass Dwell) 94.0 50 178.0 50	(Mass Dwell) 101.0 50 179.0 50	(Mass Dwell) 142.0 50 188.0 50

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	202.0 50 284.0 50	264.0 50	266.0 50
Group 4 Group Start Time: 10.50 Plot 1 Ion: 228 Ions/Dwell in Group	(Mass Dwell) 120.0 50 240.0 50	(Mass Dwell) 228.0 50	(Mass Dwell) 229.0 50
Group 5 Group Start Time: 12.00 Plot 1 Ion: 252.0 Ions/Dwell in Group	(Mass Dwell) 138.0 50 253.0 50 267.0 50	(Mass Dwell) 139.0 50 260.0 50 276.0 50	(Mass Dwell) 252.0 50 264.0 50 278.0 50

- **10.1.5.** The above listed instrument conditions are used for all analytical standards for calibration and for all sample extracts analyzed by this method.
  - **10.1.5.1** The column conditions, scan start time, and splitless valve time for analysis of DFTPP only are as follows are as follows:

Initial Column Temperature and Hold Time: 140 <sup>o</sup> C for 0.5 minutes
Column Temperature Program: 140 <sup>0</sup> to 320 <sup>0</sup> C at 22 <sup>0</sup> C/minute
Final Column Temperature Hold: 320C for 0.5 minutes
Scan Start Time: approx. 5 minutes
Splitless Valve Time: 0.25 minutes
Injection Volume: 2 ul

### 10.2. Analytical Sequence

- **10.2.1. Screening:** All samples extracts must be screened by GC/FID using the identical chromatographic conditions described in section 9.2. Screening is used to determine the dilution factor of the sample (if any) prior to GC/MS analysis (for additional details see TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision).
  - **10.2.1.1. Aqueous samples**: Prior to extract screening, the extract is diluted to 2ml and split into two 1-ml aliquots:
    - One 1-ml aliquot is internal standardized with 20ul of the 2000 ng/ul internal standard solution for full scan analysis and is analyzed by GC/FID for screening.

- The other aliquot is archived for SIM analysis which is internal standardized with 20ul of 50ppm SIM Internal Standard
- **10.2.1.2. Soil samples**: Final volume is 1ml and extracts are internal standardized with 20ul of the 2000 ng/ul internal standard solution and analyzed by GC/FID for screening.
- **10.2.1.3.** After screening analysis, the chromatogram is evaluated for high concentrations of organics. Determine dilutions by comparing the peak heights of compounds in the sample with the internal standard. The ratio of naturally present compounds to internal standards must be <5:1.
- **10.2.1.4.** Dilutions are made based on the screening analysis and prior to GC/MS analysis. Dilutions are made in 1-ml vials using microsyringes. Calculate the dilution factor using the equation below:

DF= Ph / 5 x ls

Where:

- DF = Dilution Factor
- Ph = Sample Peak Height
- Is = Internal Standard Peak Height

When DF >1 but <2, combine 500ul of sample extract with 500ul methylene chloride in a 1 ml amber vial, add20 ul internal standard and crimp seal

Use **Table 7** to determine dilution and internal standard amount.

## **10.2.2.** Instrument Performance and Calibration Sequence

- **10.2.2.1.** Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.
- **10.2.2.2.** Analyze the Instrument Performance Check Standard (DFTPP) as discussed in Section 9.2.1.
- **10.2.2.3.** Initially and as required, analyze the five (5) point Initial Calibration Range (six points for second order regression) as detailed in Sections 7.2.1 and 9.2.4.2. Evaluate the acceptability of the Initial Calibration Range as detailed in Section 9.2.4.2.

- **10.2.2.4.** Immediately after the Initial Calibration Range only, analyze the Initial Calibration Verification (ICV) as detailed in Sections 7.2.1.2 and 9.2.4.3. Evaluate the acceptability of the ICV as detailed in Section 9.2.4.3.
- **10.2.2.5.** Every 12 hours, reanalyze and evaluate the Instrument Performance Check Standard (DFTPP) followed by the Continuing Calibration Verification (CCV) as detailed in Section 9.2.3 and 9.2.4.4. Evaluate the acceptability of the CCV as detailed in Section 9.2.4.4
- **10.2.2.6.** Client samples and QC samples are analyzed (as detailed in Section 10.2.3) after acceptable Instrument Performance and Calibration Checks and until the 12 hour clock expires. Repeat the sequence as required. The automation of GC/MS runs is accomplished via the "SEQUENCE" macro of the ChemStation.

## 10.2.3. Sample Analysis Sequence

- **10.2.3.1.** Sample extracts are normally prepared on the same day as analysis. The GC/MS operator will prepare the extracts that will be run on his or her instrument. Volume adjustments to the extracts will be made at the discretion of the supervisor.
- **10.2.3.2.** Prior to the start of sample analysis the GC/MS operator will generate a sequence program containing the list of the sample extracts to be analyzed, the position on the autosampler tray, and the proper acquisition and tune methods that are to be used. This sequence program contains all the necessary information on the samples to be analyzed and how the GC/MS system is to analyze them. The sample extracts are loaded onto the autosampler (ALS) tray. Their position is verified by checking them against the ALS number on the sequence. This batch analysis will be performed automatically over the 12-hour period.
- **10.2.3.3.** The analytical run log is printed as a record of samples analyzed. The analyst will annotate the run log with any required information regarding anomalies or unusual events. The run log must be signed by the analyst and a reviewed and signed by a trained peer or manager

# 10.3. Data Processing

**10.3.1.** Prior to processing any standards or samples, target compound lists and sublists must be assembled in the Target system. These lists are required for processing of all data files including calibration files. The data includes compound names, retention time data, quantitation ions, qualitative identification ions, and the assigned internal standard for qualitative and quantitative identification.

- **10.3.2.** Key data is manually entered the first time a compound list is used for data processing. Processing data using a compound list automatically generates response factor data and updates retention information.
- **10.3.3.** The characteristic ions for target compounds, surrogate compounds, and internal standards which can be determined using SW8270C are listed in Table 8.
- **10.4. Interpretation and Qualitative Identification:** Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:
  - **10.4.1 Target Analytes:** Qualitative identification of target compounds is based on retention time and mass spectral comparison with characteristic ions in the target compound list. The reference mass spectrum is taken from a standard of the target compound analyzed by this method. The characteristic ions are the three ions of greatest relative intensity or any ions over 30% relative intensity if less than three such ions occur in the reference spectrum. Compounds are identified as present when the following criteria are met:
    - **10.4.1.1.** Once the GC/MS instrument has been setup and maintained as detailed in Section 10.1, the first operations to be performed are the performance checks and calibration standards.
    - **10.4.1.2.** The intensities of the characteristic ions of a compound maximize in the same scan or within one scan of each other.
    - **10.4.1.3.** The relative retention time (RRT) of the sample component is within  $\pm$  0.06 RRT units of the RRT of the standard component.
    - **10.4.1.4.** The most abundant ion in the standard target spectrum that equals 100% MUST also be present in the sample target spectrum.
    - **10.4.1.5.** All other ions that are greater than 10% in the standard target spectra should also be present in the sample.
    - **10.4.1.6.** The relative intensities of the characteristic ions agree within 30% of the relative intensities of these ions in the reference spectrum. (Example: For an ion with an abundance of 50% in the reference spectrum, the corresponding abundance in a sample spectrum can range between 20% and 80%).

- **10.4.1.7.** If the compound does not meet all of the criteria listed above, but is deemed a match in the technical judgment of the mass spectral interpretation specialist, the compound will be positively identified and reported with documentation of the identification noted in the raw data record.
- **10.4.2** Non-Target Analytes: Upon client request a library search to identify nontarget Tentatively Identified Compounds (TIC) is performed. The NIST/EPA/NIH mass spectral library is used to identify non-target compounds (not including internal standard and surrogate compounds) of greatest apparent concentration by a forward search of the library. The following guidelines are used by the analyst when making TIC identifications:
  - **10.4.2.1.** Relative intensities of major ions in the reference spectrum (ions greater than 10% of the most abundant ion) should be present in the sample spectrum.
  - **10.4.2.2.** The relative intensities of the major ions should agree within  $\pm 20\%$ . (Example: For an ion with an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30 and 70%).
  - **10.4.2.3.** Molecular ions present in the reference spectrum should be present in the sample spectrum.
  - **10.4.2.4.** lons present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of co-eluting compounds.
  - **10.4.2.5.** Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the sample spectrum because of background contamination or co-eluting peaks. Data system library reduction programs can sometimes create these discrepancies.
  - **10.4.2.6.** If, in the technical judgement of the mass spectral interpretation specialist, no tentative identification can be made, the compound will be reported as 'Unknown'. If the compound can be further classified the analyst may do so (i.e, 'Unknown hydrocarbon', 'Unknown acid', etc.).

## 10.5. Data Reporting

**10.5.1.** Final Report. The Target system automatically produces a data report consisting of hardcopy reports corresponding to specific data reporting requirements, which is uploaded to the TALS LIMS System for the report production group.

- **10.5.1.1.** Total Ion Chromatogram. Full length chromatogram depicting the full length of the GC/MS acquisition.
- **10.5.1.2.** Spectra of all detected target compounds. A page for each detected target compound spectra with a standard reference spectrum for comparison.
- **10.5.1.3.** The calculations of the concentrations of each target compound in the sample, reported in units of ppb, ug/kg or ug/l.
- **10.5.1.4.** Data summaries for each method blank indicating which samples were extracted with the indicated blank.
- **10.5.1.5.** A copy of the initial calibration range together with the calibration verification report, and tune report.
- **10.5.1.6.** Quality Control (QC) data report for each batch including surrogate recoveries, internal standard area summaries, LCS, MS/MSD and RPD summaries.
- **10.6.** The low-level calibration standard establishes the reporting limit. All reported data must be at a concentration at or above the low concentration standard. Any quantitative values below the report limit must be qualified as estimated.

## 11.0. <u>Calculations/Data Reduction</u>

- **11.1. Target Compounds:** are quantitated using the internal standard method (see the formula in Section 11.3).
  - **11.1.1.** Identified target compounds are quantitated using the integrated abundance from the EICP of the primary characteristic ion. The internal standard used shall be the one nearest the retention time of the analyte).
  - **11.1.2.** The average response factor (RRF) from the initial calibration is used to calculate the target analyte concentration in client samples using the formula found in Section 11.3. See Section 9.2.4 for discussion of RRF.
  - **11.1.3.** Secondary ion quantitation is utilized only when there are sample interferences preventing use of the primary characteristic ion. If secondary ion quantitation is used an average relative response factor (RRF) must be calculated using that secondary ion.
- **11.2.** Non-Target Compounds (Tentatively Identified Compounds): An estimated concentration for non-target (tentatively identified compounds) is calculated using the internal standard method (see formula in Section 11.3). For quantitation, the nearest eluting internal standard free of interferences is used. The procedure used for calculating the concentration of non-target compounds is the same as that used for target compounds (see Section 10.5.1) with the following revisions:

- **11.2.1.** The total area count of the non-target compound is used for As (instead of the area of a characteristic ion).
- **11.2.2.** The total area count of the chosen internal standard is used as Ais (instead of the area of a characteristic ion).
- **11.2.3.** A RF on 1.0 is assumed.
- **11.2.4.** The resulting concentration is qualified as estimated ('J') indicating the quantitative uncertainties of the reported concentration.
- **11.3.** Internal Standard Calculation:
  - 11.3.1. Aqueous Samples

Concentration (
$$\mu$$
g/L) = 
$$\frac{(As)(Cis)(D)}{(Ais)(RF)(Vs) (Vi) (1000)}$$

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted prior to analysis. If no dilution is performed, $D = 1$ .
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal standard
RF	=	Average response factor from the initial calibration.
Vs	=	Volume of sample extracted (ml)

The 1000 in the denominator represents the number of ul in 1 ml.

### 11.3.2. Solid Samples

Concentration (
$$\mu$$
g/KG) =  $\frac{(As)(Cis)(D)(Vt)}{(Ais)(RF)(Ws) (Vi) (1000)}$ 

Where:

As	=	Area of the characteristic ion for the target analyte in the sample
Cis	=	Concentration of the internal standard (ug/L)
D	=	Dilution factor, if the sample or extract was diluted
		prior to analysis. If no dilution is performed, D = 1.
Vi	=	Volume of the extract injected (ul)
Ais	=	Area of the characteristic for the associated internal
		standard

RF	=	Average response factor from the initial calibration.
Vt	=	Volume of concentrated extract (ul)
Ws	=	Weight of sample (g)

The 1000 in the denominator represents the number of ul in 1 ml.

**11.4.** Relative Response Factors

$$RRF = \underline{A_x} \times \underline{C_{is}} \\ A_{is} \quad C_x$$

Where:

A<sub>x</sub> = Area characteristic ion for the compound (see Table 1)
 Ais = Area characteristic ion of associated internal standard (See Table 2)
 Cis = Concentration of internal standard
 Cx = Concentration of compound in standard

**11.5.** Percent Relative Standard Deviation (% RSD) : as discussed in Section 9.2.4.2.1 (Initial calibration):

% RSD = <u>Standard Deviation of RRFs</u> Mean RRF

**11.6.** Percent Difference (% D):as discussed in Section 9.2.4.4 (Continuing calibration):

% D = 
$$\underline{RRF_c} - \overline{RRF_i} X$$
 100  
 $\underline{RRF_i}$ 

Where: RRFc = RRF from continuing calibration

 $\overrightarrow{RRF}_i$  = Mean RRF from current initial calibration **11.7.** Percent Recovery (% R): Surrogates and Spikes

Concentration (or amount) added

**11.8.** Dry Weight Correction: All solid samples must be corrected for dry weight using the following formula for dry weight determination.

Where	:	
DW	=	Percent % Dry Weight
Gd	=	Dry weight of selected sample aliquot
Gw	=	Wet weight of selected sample aliquot

Multiply the DW value times the wet weight of the sample extracted. <u>NOTE</u>: This calculation can also be performed automatically by the target system provided the DW value is available and entered into the system.

# 12.0. Method Performance

# 12.1. <u>Method Detection Limit Study (MDL)</u>

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

# 12.2. Demonstration of Capabilities

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

## 12.3. Training Requirements

Refer to TestAmerica SOP No. ED-GEN-022, (*Training*), for the laboratory's training program.

# 13.0. Pollution Control

**13.1** It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

## 14.0. Waste Management

14.1 Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica Edison SOPs Nos. ED-SPM-007 (*Disposal of Samples and Associated Laboratory Waste, current revision*) and ED-SPM-008 (*Laboratory Waste Disposal Procedures, current revision*). The following waste streams are produced when this method is carried out:

• Auto sampler vials and expired standards: These vials are collected in satellite accumulation within the instrument laboratory. The vials are then placed into a 55 steel open top drum in the waste room. When the drums are full, the drum will be collected by the waste vendor for disposal. This waste is treated for incineration.

Teris Profile Number: 50016652 Onyx Profile WIP Number: 282493

 Mixed Solvent Waste: Mixed solvent waste is collected in a small beaker inside the bench top hood. This waste is then transferred into the satellite accumulation container in the Organic Prep. Lab. on a daily basis. This material is transferred into 5 gallon solvent cans as satellite accumulation. These cans are emptied every 24 hours into a steel drum in the waste room. This drum is kept in the walk in hood until it is full. The full drum is then removed from the hood and placed on secondary containment in the waste room.

Teris Profile Number: 50016624 Onyx Profile WIP Number: 545240

## 14.1. Pollution Prevention

- **14.2.1.** Pollution prevention encompasses any technique that reduces or eliminates the quantity or toxicity of waste at the point of generation. Numerous opportunities for pollution prevention exist in laboratory operation. The USEPA has established a prevention hierarchy of environmental management techniques that places pollution prevention as the management option of first choice. Whenever feasible, laboratory personnel should use pollution prevention techniques to address their waste generation. When wastes cannot be feasibly reduced at the source, the agency recommends recycling as the next best option.
- **14.2.2.** The quantity of chemical purchased should be based on expected usage during its shelf life and disposal cost of unused material. Actual reagent preparation volumes should reflect anticipated usage and reagent stability.

## 15.0. <u>References / Cross-References</u>

**15.1.** United States Environmental Protection Agency, "Method SW8270C, Semivolatile Organic Compounds by Gas Chromatography/Mass Spectrometry", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.

- **15.2.** United States Environmental Protection Agency, "Method SW8000B: Determinative Chromatographic Separations", Test Methods for Evaluating Solid Wastes, SW846 Third Edition, Volume 1B: Laboratory Manual, Physical/Chemical Methods, Revision 3, December 1996.
- **15.3.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, current revision.
- **15.4.** TestAmerica Edison SOP No. ED-ORP-002, *SW846 Method 3510C-Extraction of Semi-Volatile Organic Compounds in Water by Separatory Funnel*, current revision.
- **15.5.** TestAmerica Edison SOP No. ED-ORP-004, *SW846 Method 3541 Automated Soxhlet Extraction of Solid-Semi-Volatile Organic Compounds,* current revision.
- **15.6.** TestAmerica Edison SOP No. ED-ORP-005, SW846 *Method 3550B- Extraction of Semi-Volatile Organic Compounds in Soil Using Low-level Extraction Technique, current revision.*
- **15.7.** TestAmerica Edison SOP No. ED-ORP-006, SW846 *Method 3550B- Extraction of Semi-Volatile Organic Compounds in Soil Using Medium--level Extraction Technique*, current revision.
- **15.8.** TestAmerica Document No. CW-E-M-001, Corporate Environmental Health and *Safety Manual,* current revision.
- **15.9.** TestAmerica Corporate Quality SOP No. CA-Q-S-001, *Solvent & Acid Lot Testing & Approval*, current revision.
- **15.10.** TestAmerica Edison SOP No. ED-GEN-023 (*Bulk Solvent Testing and Approval*), current revision.
- **15.11.** TestAmerica Edison SOP No. ED-GCS-001, *Preparation and Screening of Semivolatile Organic Extracts for GC/MS Analysis*, current revision.
- **15.12.** TestAmerica Edison Work Instruction Document No. EDS-WI-012, *Client Complaint/Corrective Action Form,* current revision.
- **15.13.** TestAmerica Edison SOP No. ED-GEN-003, *Standard Operating Procedure for Control of Non-Conformances and Corrective Action,* current revision.
- **15.14.** TestAmerica Edison SOP No. ED-ORP-001, *Extraction of Semivolatile Organic Compounds in Water, EPA Method 625,* current revision.
- **15.15.** TestAmerica Edison SOP No. ED-GEN-022, *Training,* current revision.

### 16.0. <u>Method Modifications:</u>

N/A

## 17.0. <u>Attachments</u>

N/A

## 18.0. <u>Revision History</u>

- Revision 11, Effective 08/04/2010
  - Section 1.1, Table 1: Added additional analytes and associated CAS numbers.
  - Section 7.2: Updated standard suppliers and catalog numbers and updated Calibration Standards prep information.
  - Table 2: Updated Working Standards preparation information.
  - $_{\odot}$  Table 8: Added additional analytes and associated ions.
- Revision 10, Effective 02/15/2010
  - Section 1.1, Table 1: Added additional analytes and associated CAS numbers.
  - Section 1.1, Table 1: Replaced *o-Toluidene CAS#* 95-69-2 with o-Toluidine CAS# 95-53-4.
  - Section 1.2: Revised to include the TALS database as the repository for current method limits (RL/MDL).
  - Section 3.0: Updated location of Definitions in Laboratory Quality Manual.
  - Section 6.1: Updated Analytical column information.
  - Section 7.2: Updated standard suppliers and catalog numbers and updated Calibration Standards prep information.
  - Section 7.2.6: revised to include option to use TALS Reagent Module for documentation of standards receipt and prep.
  - Section 9.0 : revised throughout to include the TALS database as the repository for current method QC limits.
  - Section 10.1.4: Updated GC/MS operating conditions (Full scan and SIM) and SIM parameters.
  - Section 10.5.1: Updated to reflect data uploaded to the TALS LIMS System for the final report.
  - Table 2: Revised to include additional standard solutions.
  - Table 8: Added additional analytes and associated ions.
  - Removed Table 9 *List of Standard Reported Spike Compounds (MS, MSD, LCS)*: no longer applies with TALS.
  - Section 15.0 (References): updated as necessary.
- Revision 9, Effective 11/6/2008
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Section 1.1: Added reference to work instruction containing current MDLs/RLs.
  - Section 1.1: Added reference to Quality Assurance Manual for method modifications.
  - Section 2: Expanded to include references to applicable prep SOPs.
  - Section 3: revised to reference new location for definitions.
  - Section 5: Updated list of potentially hazardous materials and added most recent corporate health and safety information.
  - Section 6: Revised to include 5975MSD as an instrument option. Updated HP

references to Agilent (HP).

- Section 6.1.5: Updated NBS Library reference to NIST.
- Section 7.2: updated all standards prep information. Added information on addition of a sixth initial calibration standard for second order regression.
- Section 7.2: added details of SIM calibration standards prep and SIM internal standards prep.
- Section 8.0: added a table detailing sample collection and holding time requirements.
- Section 9.0: added a table summarizing QC sample types, frequencies and limits. Added text detailing analysis and evaluation of each.
- Section 10.0: added additional detail on instrument startup and maintenance.
- Section 10.0: added detail on extract prep and screening. Added reference to screening SOP.
- Section 10.0: updated and expanded analytical sequence info, qualitative and quantitative ID info, and non-target ID info.
- Section 11.0: Added applicable calculations.
- Section12.0: added reference to Training SOP.
- References: Expanded to include more specific SOP references.
- Tables: renumbered and reformatted. Revised Table 2 to include second source ICV information. Added Table 9 (List of Standard Reported Spike Compounds)
- Revision 8, Effective 05/12/2007
  - Section 10.5.2.10.1: The last sentence in this section was revised to read as follows (the r value was previously incorrectly listed as ≤0.99):

"The r-value (Correlation Coefficient) of the equation must be  $\geq$  0.99 for the calibration to be employed."

• Section 12.3.2: Revised to read as follows (previously required re-extraction if any single surrogate recovered outside limits):

*"If any two or more surrogates for any one fraction (base-neutral or acid) are outside of recovery limits or if any one surrogate recovers at <10%, reextract the sample. If a corrective action is necessary."* 

• Section 16.2.1: Revised to read as follows:

"Samples which have any two or more surrogates for any one fraction (baseneutral or acid) outside of recovery limits or if any one surrogate recovers at <10%, reextract (volume permitting) and reanalyze the sample. If a surrogate is diluted to a concentration below that of the lowest calibration standard, no corrective action is necessary."

Table 2							
Working Standards Preparation							
Solution Name	5 PPM	10 PPM	20 PPM	50 PPM	80 PPM	120PPM	100PPM (ICV 2 <sup>nd</sup> Source)*
BN Cal Mix 1	100ul	-	-	-	-	-	-
BN Cal Mix 2	-	50ul	100ul	250ul	400ul	600ul	1000ul
3,3'-Dichlorobenzidine	20ul	40ul	60ul	100ul	160ul	240ul	4000ul
Benzidine	20ul	40ul	60ul	100ul	160ul	240ul	4000ul
N-Nitrosodiphenylamine	10ul	20ul	40ul	100ul	160ul	240ul	4000ul
OLM04 SV Mix	25ul	50ul	100ul	250ul	400ul	600ul	10000ul
AC Cal Mix 1	75ul	100ul	150ul	250ul	400ul	600ul	10000ul
AC Cal Mix 2	25ul	50ul	100ul	250ul	400ul	600ul	10000ul
1,2,3,4-TCDD	-	-	-	100ul	-	-	-
AC Surr	25ul	50ul	100ul	250ul	400ul	600ul	-
BN Surr	25ul	50ul	100ul	250ul	400ul	600ul	-
1,2,4,5-Tetrachlorobenzene	50ul	100ul	200ul	500ul	800ul	1200ul	-
2,3,4,6-Tetrachlorophenol	50ul	100ul	200ul	500ul	800ul	1200ul	-
SPEX Custom Mix	25ul	50ul	100ul	250ul	400ul	600ul	-
SPEX Special Mix	25ul	50ul	100ul	250ul	400ul	600ul	-
ISTD	200ul	200ul	200ul	200ul	200ul	200ul	-
Final Volume (ml)	10	10	10	10	10	10	200

\* The ICV is prepared using the separate source standard solutions detailed in Section 7.2.1. The ICV working standard concentration is 25 mg/l.

Table 3           Working Standards Preparation (SIM)						
	Level 1	Level 2	Level 3	Level 4	Level 5	Level 6
Pentachlorophenol	10uL	25uL	50uL	50uL	100uL	250uL
n-	10uL	25uL	50uL	50uL	100uL	250uL
Nitrosodimethylamine						
PAH mix	2.5uL	5uL	100uL	25uL	50uL	100uL
Hexachlorobenzene	10uL	25uL	100uL	500uL	1000uL	2500uL
ISTD	200uL	200uL	200uL	100uL	100uL	100uL
Final Volume (ml)	10	10	10	5	5	5

Table 4				
System Performance Monitoring Compounds (SPCC)				
N-nitroso-di-n-propylamine				
2,4-Dinitrophenol				
Hexachlorocyclopentadiene				
4-Nitrophenol				

TABLE 5 CALIBRATION CHECK COMPOUNDS (CCC)			
Base/Neutral Fraction	Acid Fraction		
Acenaphthene	4-Chloro-3-methylphenol		
1,4-Dichlorobenzene	2,4-Dichlorophenol		
Hexachlorobutadiene	2-Nitrophenol		
N-Nitrosodiphenylamine	Phenol		
Di-n-octyl phthalate	Pentachlorophenol		
Fluoranthene	2,4,6-Trichlorophenol		
Benzo(a)pyrene			

Table 6 0.1ppm SIM ICV preparation			
Pentachlorophenol	25uL		
n-Nitrosodimethylamine	25uL		
PAH mix	5uL		
Hexachlorobenzene	5uL		
ISTD	100uL		
Final Volume	5ml		

Table 7 Dilution Factor Calculations					
DF Value	Sample Amt. (ul)	CH <sub>2</sub> Cl <sub>2</sub> Amt. (ul)	ISTD Sol. Amt. (ul)		
<1	1,000 500	None 500	None 10		
>1, <2 >4, <5 >10, <20	200 100	800 800 900	16 16 36		
>20	500*	500 500	10		

\*Prepare this dilution by serially diluting the >10, <20 dilution

Table 8					
Characteristic lons Of Semi-Volatile Organic Compounds Compound Primary Ion Secondary Ion(s)					
Compound	154	Secondary Ion(s)			
Acenaphthene		153, 152			
Acenaphthylene	152	151, 153			
Anthracene	178	176, 179			
Atrazine	200	173,215			
Benzaldehyde	77	105,106			
Benzidine	184	92, 185			
Benzo(a)anthracene	228	229, 226			
Benzo(b)fluoranthene	252	253, 125			
Benzo(k)fluoranthene	252	253, 125			
Benzo(g,h,i)perylene	276	138, 277			
Benzo(a)pyrene	252	253, 125			
Biphenyl (1,1')	154	153,76			
Bis(2-chloroethoxy)methane	93	95, 123			
Bis(2-chloroethyl)ether	93	63, 95			
Bis(2-chloroisopropyl)ether	45	77, 121			
Bis(2-ethylhexyl)phthalate	149	167, 279			
4-Bromophenyl phenyl ether	248	250, 141			
Butyl benzyl phthalate	149	91, 206			
Caprolactam	113	55,56			
2-Chloronaphthalene	162	127, 164			
4-Chloro-3-methylphenol	107	144, 142			
2-Chlorophenol	128	64, 130			
4-Chlorophenyl phenyl ether	204	206, 141			
Chrysene	228	226, 229			

Table 8           Characteristic Ions Of Semi-Volatile Organic Compounds			
Compound	Primary Ion	Secondary Ion(s)	
Dibenz(a,h)anthracene	278	139, 279	
Di-n-butylphthalate	149	150, 104	
1,3-Dichlorobenzene	146	148, 111	
1,4-Dichlorobenzene	146	148, 111	
1,2-Dichlorobenzene	146	148, 111	
3,3'-Dichlorobenzidine	252	254, 126	
2,4-Dichlorophenol	162	164, 98	
Diethylphthalate	149	177, 150	
2,4-Dimethylphenol	122	107, 121	
Dimethylphthalate	163	194, 164	
4,6-Dinitro-2-methylphenol	198	51, 105	
2,4-Dinitrophenol	184	63, 154	
2,4-Dinitrotoluene	165	63, 89	
2,6-Dinitrotoluene	165	63, 89	
1,2-Diphenylhydrazine	77	105, 182	
Di-n-octylphthalate	149	167, 43	
Fluoranthene	202	101, 203	
Fluorene	166	165, 167	
2-Fluorobiphenyl (SURR.)	172	171	
2-Fluorophenol (SURR.)	112	64	
Hexachlorobenzene	284	142, 249	
Hexachlorobutadiene	225	223, 227	
Hexachlorocyclopentadiene	237	235, 272	
Hexachloroethane	117	201, 199	
Indeno(1,2,3-cd)pyrene	276	138, 227	
Isophorone	82	95,138	
Naphthalene	128	129, 127	
Nitrobenzene	77	123, 65	
	143	115, 116	
1-Naphthylamine			
2-Naphthylamine	143	115, 116	
Nitrobenzene-d₅ (SURR.)	82	128, 54	
2-Nitrophenol	139	109, 65	
4-Nitrophenol	139	109, 65	
N-Nitrosodimethylamine	42	74, 44	
N-Nitrosodiphenylamine	169	168, 167	
N-Nitroso-di-n-propylamine	170	42,101,130	
o-Toluidine	107	106, 77	
Pentachlorophenol	266	264, 268	
Phenanthrene	178	179, 176	
Phenol	94	65, 66	
Phenol-d₅ (SURR. )	99	42, 71	
Pyrene	202	200, 203	
Terphenyl-d <sub>14</sub> (SURR.)	244	122, 212	
2,4,6-Tribromophenol (SURR.)	330	132, 141	
1,2,4-Trichlorobenzene	180	182, 145	
2,4,6-Trichlorophenol	196	198, 200	

Table 8           Characteristic Ions Of Semi-Volatile Organic Compounds			
Compound	Primary Ion	Secondary Ion(s)	
1,4-Dichlorobenzene d <sub>4</sub> (I.S.)	152	150, 115	
Naphthalene $d_8$ (I.S.)	136	68	
Acenaphthene $d_{10}$ (I.S.)	164	162, 160	
Phenanthrene d <sub>10</sub> (I.S.)	188	94, 80	
Chrysene d <sub>12</sub> (I.S.)	240	120, 136	
Perylene d <sub>12</sub> (I.S.)	264	260, 265	
n-decane	43	57	
2-tert-butyl-4-Methylphenol	149	121, 91	
3,5-Di-tert-butyl-4-Hydroxytol	205	220, 145	
Coumarin	146	118, 63	
n-Octadecane	57	43, 85	
2-Methylnaphthalene	142	141	
1-Methylnaphthalene	142	141, 115	
1,3-Dimethylnaphthalene	156	141, 115	
Carbamazepine	193	236, 135	
N,N-Dimethylaniline	120	122, 104	
Diphenyl Ether	170	77, 115	
1,4-Dioxane	88	58, 43	
Pyridine	79	52, 51	
Aniline	93	66	
Benzyl Alcohol	108	79, 77	
2-Methylphenol	108	107	
Acetophenone	105	77, 51	
4-Methylphenol	108	107	
Benzoic Acid	122	105, 77	
4-Chloroaniline	127	129	
2,4,5-Trichlorophenol	196	198, 200	
2-Nitroaniline	65	108, 138	
3-Nitroaniline	138	108, 65	
4-Nitroaniline	138	108, 65	
Dibenzofuran	168	139	
Carbazole	167	166, 139	
2,3,7,8-TCDD (screen)	320	322, 324	
1,2,4,5-Tetrachlorobenzene	216	214, 179	
2,3,4,6-Tetrachlorophenol	232	131, 230	



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# Title: Analysis of Total and Amenable Cyanide in Water, Drinking water, Wastewater, Soil, and Wipes- Automated by Method EPA SW846 9012A/9012B; 335.4 and Standard Method 4500 CN<sup>-</sup>C

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#### 1.0 Scope and Application

#### 1.1 <u>Analytes, Matrix(s), and Reporting Limits</u>

- **1.1.1** Methods EPA 335.4, SM 4500 CN<sup>-</sup>C, SW846 9012A/9012B, and Lachat 10-204-00-1-A are described in this SOP. The SOP is applicable to water, drinking water, wastewater, soil, waste, and wipe samples requiring cyanide determination. The method detects inorganic cyanides that are present as either soluble salts or complexes. It is used to determine values for both total cyanide, free cyanide and cyanide amenable to chlorination.
- **1.1.2** The automated method has a detector that is sensitive to approximately to 0.005 mg CN-/L. The laboratory's reporting limit for water sample is 0.01 mg/L and the reporting limit for soil sample is 0.50 mg/kg. The reporting limit for wipes is 0.0005 mg/wipe.
- **1.1.3** On occasion clients may request modifications to this SOP. These modifications are handled following the procedures outlined in Sections 7 (Review of Work Request) and 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 2.0 <u>Summary of Method</u>

Cyanide is extracted by means of reflux distilliation (EASY-Dist distillation apparatus). Sample is distilled in a strongly acidic solution which breaks down cyanide complexes and converts the cyanide to hydrocyanic acid (HCN). HCN is absorbed into a sodium hydroxide absorber solution and the cyanide ion in the absorbing solution is determined by automated UV colorimetry at 570nm.

#### 3.0 <u>Definitions</u>

For a complete list of definitions refer to Appendix 2 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 4.0 Interferences

- **4.1** Interferences can be reduced or eliminated by using the distillation procedure.
- **4.2** The presence of sulfide in the sample matrix adversely effects the determination of total cyanide. Samples that contain hydrogen sulfide, metals sulfides or other compounds that may produce hydrogen sulfide during distillation should be treated.
  - **4.2.1** Place a drop of the sample on lead acetate test paper (which has been premoistened with pH 4 acetate buffer solution) to detect the presence of sulfides. If sulfides are present (test strip turns black), the sample volume required for the cyanide determination should be increased by 25 milliliters (ml).

- **4.2.2** The total volume of sample should be treated with powdered cadmium carbonate or lead carbonate. Yellow cadmium sulfide precipitates if the sample contains sulfide.
- **4.2.3** Repeat this operation until a drop of the treated sample solution does not darken the lead acetate test paper
- **4.2.4** Filter the solution through a dry filter paper into a dry beaker, and from the filtrate measure the sample to be used for analysis.

NOTE: Avoid a large excess of cadmium carbonate and a long contact time in order to minimize a loss of cyanide on the precipitated material.

- **4.3** Oxidizing agents such as chlorine decompose most cyanides.
  - **4.3.1** To determine if oxidizing agents are present test a drop of sample with potassium iodine-starch paper. If a blue color appears that indicates the need for treatment. Add ascorbic acid, a few crystals at a time, to the sample until a drop of sample produces no blue color on the indicator paper. Add an additional 0.6g of ascorbic acid for each liter of sample volume.
- **4.4** Nitrates and nitrites may give high biased results. During distillation they form nitrous acid, which will react with some organic compounds to form oximes. These compounds once formed will decompose to generate HCN. They are interferences when present above 10 mg/L with other specific organic compounds.
  - **4.4.1** Nitrate and nitrite interferences are eliminated by treatment with sulfamic acid just before distillation.
- **4.5** Thiocyanate is reported to be an interference when present at very high levels. Levels of 10 mg/L were not found to interfere.
- **4.6** Fatty acids, detergents, surfactants may cause foaming during the distillation process, making the endpoint difficult to detect. If this occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.

# 5.0 <u>Safety</u>

Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual (CW-E-M-001) and this document. This procedure may involve hazardous material, operations and equipment. This SOP does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of the method to follow appropriate safety, waste disposal and health practices under the assumption that all samples and reagents are potentially hazardous. Safety glasses, gloves, lab coats and closed-toe, nonabsorbent shoes are a minimum.

### 5.1. Specific Safety Concerns or Requirements

Ensure cooling water is turned on to the distillation unit. Otherwise the samples may boil over and come into contact with the heating plates. Potassium Cyanide will give off Hydrogen Cyanide (HCN) gas if combined with strong acids. Inhalation of CN gas can cause irritation, dizziness, nausea, unconsciousness and potentially death.

#### 5.2. Primary Materials Used

The following is a list of the materials used in this method, which have a serious or significant hazard rating. Note: This list does not include all materials used in the method. The table contains a summary of the primary hazards listed in the MSDS for each of the materials listed in the table. A complete list of materials used in the method can be found in the reagents and materials section. Employees must review the information in the MSDS for each material before using it for the first time or when there are major changes to the MSDS.

Material (1)	Hazards	Exposure Limit (2)	Signs and symptoms of exposure
Phosphoric Acid	Corrosive	1 Mg/M3 TWA	Inhalation is not an expected hazard unless misted or heated to high temperatures. May cause redness, pain, and severe skin burns. May cause redness, pain, blurred vision, eye burns, and permanent eye damage.
Sodium Hydroxide	Corrosive	2 Mg/M3- Ceiling	Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Contact with skin can cause irritation or severe burns and scarring with greater exposures. Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.
Pyridine	Flammable Irritant	5 ppm-TWA	Inhalation causes severe irritation to the respiratory tract. Symptoms of overexposure include headache, dizziness, nausea, and shortness of breath. Causes severe irritation possibly burns, to the skin. Symptoms include redness and severe pain. Absorption through the skin may occur, resulting in toxic effects similar to inhalation. May act as a photosensitizer. Vapors cause eye irritation. Splashes cause severe irritation, possible corneal burns and eye damage.
Barbituric Acid	Irritant	Not established	Limited information. Inhalation may irritate respiratory tract. Causes skin and eye irritation. Should be treated as potential health hazard; do not ingest.
Hydrochloric Acid	Corrosive Poison	5 ppm – Ceiling	Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and upper respiratory tract, and in severe cases, pulmonary edema, circulatory failure, and death. Can cause redness, pain, and severe skin burns. Vapors are irritating and may cause damage to the eyes. Contact may cause severe burns and permanent eye damage.

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Material (1)	Hazards	Exposure	Signs and symptoms of exposure
		Limit (2)	
Potassium Hydroxide	Poison Corrosive Reactive	2 mg/m3 – Ceiling	Inhalation symptoms may include coughing, sneezing, damage to the nasal or respiratory tract. High concentrations can cause lung damage. Swallowing may cause severe burns of mouth, throat and stomach. Other symptoms may include vomiting, diarrhea. Severe scarring of tissue and death may result. Contact with skin can cause irritation or severe burns and scarring. Causes irritation of eyes with tearing, redness, swelling. Greater exposures cause severe burns with possible blindness.
Potassium Cyanide	Poison Corrosive	5 Mg/M3 TWA as CN	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Sodium Cyanide	Poison Corrosive	5 mg/m3 TWA as CN (skin)	This material will form Hydrogen Cyanide (HCN) gas when combined with strong acids. Breathing HCN gas may result in death. Corrosive to the respiratory tract. May cause headache, weakness, dizziness, labored breathing nausea and vomiting, which can be followed by weak and irregular heartbeat, unconsciousness, convulsions, coma and death. Solutions are corrosive to the skin and eyes, and may cause deep ulcers, which heal slowly. May be absorbed through the skin, with symptoms similar to those noted for inhalation. Symptoms may include redness, pain, blurred vision, and eye damage.
Silver Nitrate	Poison Corrosive Oxidizer	0.01mg/m3 (TWA) for silver metal dust and fume as Ag	Inhalation symptoms may include burning sensation, coughing, wheezing, laryngitis, shortness of breath, headache, nausea, and vomiting. May be absorbed into the body following inhalation. Swallowing can cause severe burns of the mouth, throat and stomach. Can cause sore throat, vomiting, and diarrhea. Poison. Symptoms include pain and burning in the mouth, blackening of the skin and mucous membranes, throat, and abdomen, salivation, vomiting of black material, diarrhea, collapse, shock, coma and death. Skin contact can cause redness, pain and severe burns. Eye contact can cause blurred vision, redness, pain, severe tissue burns and eye damage.
1 – Always add a	cid to water to p	revent violent rea	
2 – Exposure limi	t refers to the O	SHA regulatory e	exposure limit.

# 6.0 Equipment and Supplies

# 6.1. Instrumentation

• EASY- Dist distillation apparatus with all associated glassware (Westco Scientific Instruments)

- Flow injection analysis equipment-designed to deliver and react sample and reagents in the required order and ratios. Lachat 8000 series
  - > Multichannel proportioning pump
  - ➤ Reaction manifold
  - ➤ Colorimetric detector
  - ≻Data system
  - ≻ Heating Unit

#### 6.2. <u>Supplies</u>

- Various Class A volumetric flask and pipettors
- Micro-porous boiling chips
- 120 ml snap seal sample cups
- 24/40 clips
- Potassium Iodine-starch paper
- Lead-Acetate paper (pre-moistened with pH 4 acetate buffer solution)
- Analytical balance
- Vacuum pump
- Specimen cups
- Transfer pipettes
- Graduated cylinder (class A)
- Micro-buret

# 7. <u>Reagents and Standards</u>

#### 7.1. Reagents

- **7.1.1.** Deionized water-18 megohm reagent grade Type II water.
- **7.1.2.** Acetone store at room temperature; for stability information refer to manufacturer's instructions.
- **7.1.3.** Rhodanine indicator p-dimethyl-aminobenzalrhodanine: Place 20 mg of pdimethyl-aminobenzalrhodanine in a 100ml volumetric flask and dilute to the

mark with acetone. Store at room temperature. Stable for six months.

- 7.1.4. Acetate Buffer Dissolve 82.0 gram of NaC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> x 3H<sub>2</sub>O in 100ml DI water. Add sufficient glacial acetic acid and adjust pH to 4.5 (approx. 10 drops). This solution is stable for 6 months, store at 4°C.
- **7.1.5.** Cadmium Carbonate (powder) ACS reagent –store at room temperature; discard after 5 years from date receipt or earlier if necessary.
- **7.1.6.** Reagents for cyanides amenable to chlorination.
  - **7.1.6.1.** Calcium hypochlorite solution, (0.35M), Ca(OCl)<sub>2</sub>. Dissolve 5 g of calcium hypochlorite in 100 ml of deionized water. Solution is stable for 6 months, store at room temperature.
  - **7.1.6.2.** 0.25 M NaOH. Dissolve 10.0 g NaOH into 1 L of deionized water. Dilute to mark with deionized water and invert to mix. Solution is stable for 6 months, store at room temperature.
  - **7.1.6.3.** Ascorbic acid,  $C_6H_8O_6$  store at room temperature, for stability information, refer to manufacturer's instructions.
- 7.1.7. Reagents for cyanide distillation.
  - 7.1.7.1. 0.25 M NaOH. See section 7.1.6.2
  - **7.1.7.2.** Sulfamic Acid Solution (0.4N): Dissolve 40.0 g. H<sub>2</sub>NSO<sub>3</sub>H into 1000ml of deionized water. Solution is stable for 6 months, store at room temperature.
  - **7.1.7.3.** Magnesium chloride solution (2.5M): Dissolve 510 gm of  $MgCl_2 x$   $6H_2O$  in 1 liter of deionized water. Solution is stable for 6 months, store at room temperature.
  - **7.1.7.4.** 1:1  $H_2SO_4$ : Slowly pour 500 ml  $H_2SO_4$  into 500 ml deionized water. (Caution! Solution is very hot.) Solution is stable for 6 months, store at room temperature.
  - **7.1.7.5.** Bismuth nitrate Solution: Dissolve 3 g of Bi(NO)<sub>3</sub>•5H<sub>2</sub>O in 10 mls DEIONIZED WATER , add 25 mls glacial acetic acid while stirring and dilute to a 100 ml final volume.
- **7.1.8.** Reagents for automated colorimetric determination.
  - 7.1.8.1. Pyridine-Barbituric acid Reagent: In a fume hood, place 15.0 g barbituric acid in a 1 L beaker and add 100 ml DEIONIZED WATER, rinsing down the sides of the beaker to wet the barbituric. Add 75 ml pyridine (C<sub>5</sub>H<sub>5</sub>N) while stirring and mix until barbituric acid dissolves. Add 15 ml conc. HCI (12 M HCI) and mix. Transfer to a 1 L volumetric flask, dilute to mark and invert to

mix. Prepare weekly, store solution in amber bottle and at room temperature.

- **7.1.8.2.** Chloramine-T: Dissolve 2.0g chloramine-T hydrate in 500 ml deionized water. Prepare fresh daily. Store at room temperature.
- **7.1.8.3.** Phosphate buffer: In a 1 L volumetric flask, dissolve 97.0 g potassium phosphate, monobasic, anhydrous, (KH<sub>2</sub>PO<sub>4</sub>) in approximately 800 ml of DEIONIZED WATER. Dilute to mark and invert to mix. Solution is stable for one month. Store at room temperature.
- **7.1.8.4.** 0.25 M NaOH (carrier) See section 7.1.6.2.

#### 7.2. Standards

- **7.2.1.** Standard 0.0192N Silver nitrate solution (1 ml = 1 mg CN): Prepared by weighing 3.2647 g of dried AgNO<sub>3</sub>, dissolved in deionized water and dilute to 1000ml. Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4°C.
- 7.2.2. Primary Cyanide stock solution, 1000 mg/L: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Standardize the solution against 0.0192N silver nitrate solution weekly and dilute to the appropriate concentration so that 1ml=1mg CN. Solution is stable for 6 months, store at 4°C. Standardize the solution as follows:
  - Take 5.0 ml of the primary stock cyanide solution (Sec. 7.2.2) and dilute it to 50ml using the 0.25 M NaOH. Add 10-12 drops of the rhodanine indicator (Sec. 7.1.3) to produce a bright yellow color.
  - Using the 10 ml micro buret, titrate the stock cyanide solution from a bright yellow color to a salmon hue endpoint. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
  - Titrate a blank using 50mls of 0.25N NaOH, 10-12 drops of rhodanine indicator. Record the volume of silver nitrate solution used for the titration in the Standardization logbook.
  - > Calculate CN concentration as follows:

Based on the following:  $Ag^+ + 2 CN \rightarrow [Ag(CN)_2]^-$ 

 $CN^{-}(mg/L) = \frac{A-B}{C} \times N \frac{2 \text{ mol CN}}{1 \text{ eq AgNO}_3} \times \frac{26.02 \text{ g CN}}{1 \text{ mol CN}} \times \frac{1000 \text{ mg}}{1 \text{ g}}$ 

Where:

A= ml AgNO<sub>3</sub> for titration of sample B=ml AgNO<sub>3</sub> for titration of blank

C= ml or g of sample N= normality of AgNO<sub>3</sub>

- Record the preparation in the reagent TALS. Record final concentration in the Standardization logbook.
- **7.2.3.** Secondary Cyanide Stock Solution, 1000mg/L: Dissolve 2.51 gm of KCN and 2.0 gm of KOH in 900 ml of deionized water. Note: the secondary cyanide stock solution must be prepared from a different source than the primary cyanide stock solution. Standardize the solution with 0.0192N silver nitrate solution monthly and dilute to the appropriate concentration so that 1ml=1mg CN. The solution is stable for 6 months, store at 4°C. Follow the standardization procedure in 7.2.2.
- **7.2.4.** Primary Working Solution, 5mg/L: Take 1ml of the Primary Cyanide Stock Solution (Sec. 7.2.2) and dilute to a final volume of 200ml with 0.25N NaOH. Prepare fresh daily.
- **7.2.5.** Primary Working Solution, 10mg/L: Take 1ml of the Primary Cyanide Stock Solution (Sec. 7.2.2) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.
- **7.2.6.** Secondary Working Solution, 10mg/L: Take 1ml of the Secondary Cyanide Stock Solution (Sec. 7.2.3) and dilute to a final volume of 100ml with 0.25N NaOH. Prepare fresh daily.

# 8. <u>Sample Collection, Preservation, Shipment and Storage</u>

Sample container, preservation techniques and holding times may vary and are dependent on sample matrix, method of choice, regulatory compliance, and/or specific contract or client requests. Listed below are the holding times and the references that include preservation requirements.

Matrix	Sample Container	Min. Sample Size	Preservation	Holding Time <sup>1</sup>	Reference
Waters	Glass or plastic	200 ml	NaOH, pH > 12; Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012A
Soils	Glass	5.0 grams	Cool 4 ± 2°C	14 Days	Method 335.4; SW846 Method 9012A
Wipes	Glass	1 wipe	Cool 4 ± 2°C	14 Days	SW846 Method 9012A

<sup>1</sup> Inclusive of digestion and analysis.

#### 9. Quality Control

- **9.1.** <u>Sample QC:</u> The following sample QC must be run with each set up of samples or every 10 samples, whichever is less. Waters, soils, and wipes are separated into different QC batches.
  - **9.1.1.** <u>Method Blank (MB):</u> Deionized water is used for the blank and is carried through the entire sample preparation and analytical process. Results must be less than the reporting limit, or the batch must be redistilled and reanalyzed. Note: For *wipe* sample batches, the method blank consists of a blank wipe plus 50 ml of deionized water.
  - **9.1.2.** <u>Laboratory Control Sample (LCS):</u> Prepare fresh daily.
    - Laboratory Control Sample-Low (LLCS), 0.025 mg/L: Add 50ml of 0.25N NaOH to the boiling tube then add 0.25 ml of the 5 mg/L CN working solution (Sec. 7.2.4). The results must be within 90-110% of the true value or the batch must be redistilled and reanalyzed.
    - Laboratory Control Sample –High (HLCS), 0.20 mg/L: Add 50ml of 0.25N NaOH to the boiling tube then add 1.0 ml of the 10 mg/L CN working solution (Sec. 7.2.5). The results must be within 90-110% of the true value or the batch must be redistilled and reanalyzed.
    - Note: For wipe batches, prepare an LCS/LCSD at 0.2 mg/L. Add a blank wipe to the boiling tube and follow the preparation procedure above.

#### 9.1.3. <u>Matrix Spike/Matrix Spike Duplicate (MS/MSD)</u>

- Aqueous samples (drinking water and wastewater): two portions of the same sample (matrix spike and matrix spike duplicate), each 50 ml, are added to separate boiling tubes then add 1.0 ml of the 10 mg/L CN primary working solution (Sec 7.2.5) into each sample; the spiking concentration is 0.20mg/L. The results must be within 90-110%.
- Soil samples: two portions of the sample (matrix spike and matrix spike duplicate) are added to separate boiling tubes and 50 ml of deionized water is added. Spike these samples with 1.0 ml of the 10 mg/L CN primary working solution (Sec 7.2.5). The results must be within laboratory generated limits.
- > Wipe samples: matrix spikes are not required.

# 9.2. Instrument QC

**9.2.1.** <u>Initial Calibration Verification (ICV), 0.20 ppm</u>: The ICV must be analyzed immediately after an acceptable calibration and before any samples are analyzed. The ICV is prepared by taking 2ml of the 10ppm secondary working solution (Sec. 7.2.6) and diluted up to a final volume of 100ml with

0.25N NaOH solution. The determined concentration must be within  $\pm 10\%$  of the true value, if not the analysis should be terminated until the source of the problem is identified and corrected.

- **9.2.2.** <u>Continuing Calibration Verification (CCV), 0.20 ppm:</u> A CCV is analyzed after every 10 samples and at the end of the sample run. The CCV is prepared the same way as the ICV. The determined concentration must be within ±10% of the true value. If the result exceeds the limit, the analysis should be terminated until the source of the problem is identified and corrected and all samples following the last acceptable CCV must be reanalyzed.
- **9.2.3.** <u>Initial Calibration Blank (ICB):</u> An ICB is analyzed immediately after the ICV to verify calibration and acceptable instrument performance. The results must be less than the reporting limit. Use 0.25M NaOH for the ICB.
- **9.2.4.** <u>Continuing Calibration Blank (CCB)</u>: A CCB must be analyzed after every 10 samples and at the end of the sample run. The results must be less than the reporting limit. If the CCB result exceeds the reporting limit, all samples following the last acceptable CCB must be reanalyzed. Use 0.25M NaOH for the CCB.

#### 10. Procedure

#### 10.1. Sample Preparation

- **10.1.1.** Each sample matrix must be spot tested for the presence of interfering ions.
  - **10.1.1.1.** Check for the presence of chlorine using KI-Starch paper. See section 4.3.1.
  - 10.1.1.2. Check for the presence of sulfide using lead acetate paper (premoistened with pH4 acetate buffer solution). See section 4.2.1.
     4.2.4. Method 9012A/9012B: If positive add bismuth nitrate to the sample. At the same time add bismuth nitrate to all standards before distillation, see Section 10.2.3.
- **10.1.2.** Procedure for the pretreatment of cyanides amenable to chlorination.
  - 10.1.2.1. Two aliquots of the sample are used to determine cyanides amenable to chlorination. Measure 50 ml of sample to a 100 ml beaker and add calcium hypochlorite solution (Section 7.1.6.1) drop-wise while agitating the mixture using a magnetic stir plate. Maintain the pH at between 11 and 12 with sodium hydroxide solution (Section 7.1.6.2)

CAUTION: Toxic cyanogen chloride is the initial reaction product during an alkaline chlorination. This reaction should take place in a fume hood.

- 10.1.2.2. Residual chlorine must be maintained in the solution for 1 hour. Monitor pH with pH test paper and chlorine with KI-starch paper. A distinct blue color on the paper indicates sufficient chlorine in solution.
- **10.1.2.3.** Following one hour, add ascorbic acid crystals on 0.5 g portions until a test with the KI-Starch paper shows no residual chlorine. Add an excess of 0.5 g of ascorbic acid crystals to insure that no residual chlorine is present as a reducing agent.
- **10.1.2.4.** Test for total cyanide in both a chlorinated and unchlorinated aliquot of the sample. Cyanide amenable to chlorination is determined by the difference between the total cyanide result of the un-chlorinated aliquot and the chlorinated aliquot.
- 10.1.3. Distillation Procedure
  - **10.1.3.1.** For *aqueous* samples, add 50 ml sample into the boiling tube. For *solid* samples, weigh 1.0 gram of sample into the boiling tube and add 50 ml deionized water. For *wipe* samples, add 1 wipe into the boiling tube, add 50 ml of deionized water and record 1 wipe as the initial amount in TALS prep batch worksheet.
  - **10.1.3.2.** Add a few micro-porous boiling chips to the boiling tube.

THE HEATER BLOCK TEMPERATURE MUST BE 70°C OR LOWER TO LOAD BOILING TUBES. DO NOT PRE-HEAT BLOCK ABOVE 70°C. Loading tubes containing sample into a hot block will cause super-heating of samples and a potential boil-over of tube contents.

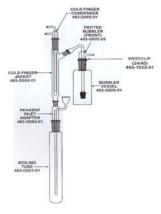


Figure 1: EASY-Dist Cyanide Distillation Apparatus

**10.1.3.3.** Add 50 mls of 0.25 M NaOH in bubbler vessel. Place the fritted bubbler (front) in the bubbler vessel and seal the joint with a size 24/40 clip. This is the NaOH absorber.

- **10.1.3.4.** Connect the reagent inlet adapter to the boiling tube. Connect the cold finger jacket to the reagent inlet adapter. Attach the cold finger condenser to the cold finger jacket.
- **10.1.3.5.** Attach the sealed NaOH absorber to the cold finger condenser. Turn the condenser water on.
- **10.1.3.6.** Put approximately 500 mls of NaOH in the excess cyanide trap.
- **10.1.3.7.** Attach the black vacuum line from the EASY-Dist gas manifold port to the inlet of the NaOH absorber.
- **10.1.3.8.** Turn vacuum on and adjust the individual air flow rate using the control knob at each manifold port. Adjust the flow such that two to five bubbles per second are exiting the base of the reagent inlet adapter. When properly adjusted, the NaOH absorber solution will be actively foaming without bubbling over into the vacuum line.
- **10.1.3.9.** If the sample is sludgy or the analyst suspects sulfide, add 5 mls bismuth nitrate solution to sample before adding the sulfamic acid.
- **10.1.3.10.** Add 5 mls sulfamic acid solution to the boiling tube through the reagent inlet adapter and let mix for three minutes.
- **10.1.3.11.** Add 5 mls 1:1 H<sub>2</sub>SO<sub>4</sub> to the boiling tube through the reagent inlet adapter. Rinse the acid down the inlet with small volume of distilled water and allow content to mix for three minutes.
- **10.1.3.12.** Add 2 mls magnesium chloride solution to the boiling tube through the reagent inlet adapter and rinse down with small volume of distilled water.
- 10.1.3.13. Turn on heating block. Heater block is set at 2.0 hour. Temperature should reach 125 +/- 3°C in approx. 20 minutes. The 1.5 hour reflux starts after the programmed temperature set point is reached. Heating block will automatically shut off after 1.5 hour.
- **10.1.3.14.** Periodically monitor the vacuum and heating to avoid excessive bubbling in the bubbler vessel. Adequate heating is indicated by a constant reflux of condensed vapor off of the condenser. Discontinue heating but maintain vacuum flow. Cool glassware apparatus for 15 minutes.
- **10.1.3.15.** Turn off vacuum pump. Remove bubbler vessel and transfer its contents into a 125ml snap seal cups. DO NOT DILUTE.
- **10.1.3.16.** The solution is now ready for semi-automated colorimetry.

#### 10.2. Calibration

**10.2.1.** Prepare fresh calibration standards everyday or before each analysis.

- 10.2.2. Preparation of Standards for samples without sulfide:
  - **10.2.2.1.** Use the 10 mg/l primary working solution (Sec. 7.2.5) to prepare the calibration standards below. Note: For wipe batches, the on-instrument concentrations are expressed in mg/L. Final results are calculated to mg/wipe when data is imported into TALS.

Volume (ml) of 10mg/L CN primary working solution	Final Volume (ml) using 0.25N NaOH	Concentration CN-(mg/L)	Concentration CN-(mg/wipe)
2.0	50	0.40	0.02
1.0	50	0.20	0.01
0.5	50	0.10	0.005
0.25	50	0.05	0.0025
0.25	100	0.025	0.00125
2.5 **	50	0.010	0.0005
0	50	0.0	0.0

\*\* For the 0.01 mg/L standard, take 2.5 ml of the 0.20 mg/L standard and dilute to 50 ml with 0.25N NaOH.

Note: Add a calibration standard of 0.005 CN mg/l as directed on special projects.

- **10.2.2.2.** Correlation coefficient of 0.995 or greater must be obtained.
- **10.2.3.** Method 9012A/9012B: Preparation of Standards for samples with sulfide:
  - **10.2.3.1.** From the 10 mg/L primary working standard prepare five standards as above to be distilled in the same manner as the samples using the method of standard additions. Note: Bismuth Nitrate must also be added to the standards. Standards distilled in this way will give a linear curve at lower concentrations but as concentration increased the recovery decreases.
  - **10.2.3.2.** Correlation coefficient of 0.995 or greater must be obtained.

#### 10.3. Sample Analysis

**10.3.1.** System startup:

- **10.3.1.1.** Follow manifold scheme for this method.
- **10.3.1.2.** Turn on heating unit at 60° C for 15 minutes before analysis.

- **10.3.1.3.** Put all reagent lines into deionized water and turn pump on. Check for leaks and slow flow rate.
- **10.3.1.4.** Switch over reagent lines to proper reagent. Let reagent run through manifold for 15 minutes before analysis to allow baseline to stabilize.
- **10.3.2.** Set up tray table with all appropriate QC checks. CCV/CCB every 10 samples and at the end of the run. The following analytical sequence must be used:

Instrument Calibration QCS (ICV) CCB IPC (CCV) Method Blank LLCS HLCS 7 Samples with Analytical Spikes IPC (CCV) CCB 10 samples IPC (CCV) CCB Repeat until run is complete IPC (CCV) CCB

Note: An LCS/LCSD will be substituted for the LLCS/HLCS in the analytical sequence for wipes.

- **10.3.3.** Place standards and samples into autosampler cups.
- **10.3.4.** Run tray.
- **10.3.5.** Dilute and reanalyze samples that exceed the calibration curve using 0.25N NaOH.
- **10.3.6.** Free Cyanide samples will be treated following the procedure for the Cyanide Amenable to chlorination (Sec 10.1.2) and therefore must be distilled.

#### 11.0. Calculations / Data Reduction

**11.1.** Accuracy:

ICV / CCV, LCS % Recovery = <u>observed concentration</u> x 100 known concentration

MS % Recovery = (spiked sample) - (unspiked sample) x 100 spiked concentration

**11.2.** Precision (RPD):

Matrix Duplicate (MD) = <u>lorig. sample value - dup. sample value</u> x 100 [(orig. sample value + dup. sample value)/2]

**11.3.** Final results calculation in aqueous samples:

Concentration = mg/L

Concentration (mg/L) from instrument is imported to TALS Samples with dilution factor are calculated in TALS for final results

**11.4.** Final results calculation in solid samples:

Concentration mg/kg =  $\frac{C \times V \times D}{W}$ Where:

= sample conc

C = sample concentration in extract (mg/L) V = Volume of extract (mL) D = Dilution Factor W = Weight/Volume of sample aliguot extracted (grams)

NOTE: All dry weight corrections are made in TALS.

**11.5.** Final results calculation in wipe samples:

Concentration mg/wipe =  $\frac{C \times V \times D}{1000}$ Where:

C= sample concentration in extract (mg/L) V=Volume of extract (mL) D=Dilution factor (if present)

#### **11.6.** Data Reduction

- **11.6.1.** Instrument data is imported to TALS by following these steps:
  - **11.6.1.1.** After the run has finished, click the data icon at the top of the screen. Choose the correct file. Go to 'File" and choose "Export Data' from the drop down menu. A screen will pop up, click 'ok' and then another screen will pop up, click 'ok.'
- **11.6.2.** From the desktop, open the LIMSdata icon. Select the correct file, and then right click. Go to 'Send to-> LimsImport ->' Choose the correct method. The data has now been imported to TALS.

- **11.6.3.** All reagent information is recorded in the "Batch Information" page. Use the 'Worksheet tab' if additional pages are necessary.
- **11.6.4.** Record special notes and observations in the "worksheet" tab (i.e. sample appearance and notes on why samples were rejected or diluted).
- **11.6.5.** All raw data is attached as a pdf file. The raw data includes the instrument report and calibration curve.
- **11.6.6.** Analyst must fill out the Wet Chem Data Review checklist (WI# EDS-WI-008) during the first level review. The batch is second level reviewed and the checklist is filed in wetchem department.

#### 12.0. Method Performance

#### 12.1. Method Detection Limit Study (MDL)

The method detection limit (MDL) is the lowest concentration that can be detected for a given analytical method and sample matrix with 99% confidence that the analyte is present. The MDL is determined according to the laboratory's MDL procedure in Section 20 (Test Methods and Method Validation) of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM). MDLs reflect a calculated (statistical) value determined under ideal laboratory conditions in a clean matrix, and may not be achievable in all environmental matrices. The laboratory maintains MDL studies for analyses performed; these are verified at least annually unless method requirements require a greater frequency.

#### 12.2. <u>Demonstration of Capabilities</u>

For DOC procedure refer to Section 20 in the most current revision of TestAmerica Edison's Quality Assurance Manual (ED-QA-LQM).

#### 12.3. <u>Training Requirements</u>

Refer to TestAmerica SOP No. ED-GEN-022, Training, for the laboratory's training program.

#### 13.0. Pollution Control

It is TestAmerica's policy to evaluate each method and look for opportunities to minimize waste generated (i.e., examine recycling options, ordering chemicals based on quantity needed, preparation of reagents based on anticipated usage and reagent stability). Employees must abide by the policies in Section 13 of the Corporate Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention."

#### 14.0. Waste Management

- **14.1.** Waste management practices are conducted consistent with all applicable rules and regulations. Excess reagents, samples and method process wastes are disposed of in an accepted manner. Waste description rules and land disposal restrictions are followed. Waste disposal procedures are incorporated by reference to TestAmerica SOP No. ED-SPM-008 (Laboratory Waste Disposal Procedures).
- **14.2.** The following waste streams are produced when this method is carried out:
  - Pyridine/Hydroxide Solution: Hazardous Waste Liquid, n.o.s. This material is collected at satellite accumulation in 30 gallon Poly containers. When the container is full, the container is transferred to the waste room and held in secondary containment until a waste vendor is called for a pick up.

Teris Profile Number: 50016717 Onyx Profile WIP Number: 584650

#### 15.0. <u>References / Cross-References</u>

- 15.1. <u>Methods for the Determination of Inorganic Substances in Environmental Samples</u>, EMSL-Cincinnati, EPA/600/R-93/100, August 1993, Method - 335.4 Determination of Total Cyanide by Semi-Automated Colorimetry
- **15.2.** <u>Standard Methods for the Examination of Water and Wastewater</u>, <u>18th Edition</u>, American Public Health Association, Baltimore Maryland, <u>1992</u>, SM 4500-CN<sup>-</sup>C.
- 15.3. <u>Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, 3rd ed.</u>, U.S. Environmental Protection Agency, Office of Solid Waste and Emergency Response. U.S. Government Printing Office: Washington, DC, 1995; SW846 Method 9012A, 9012B.
- **15.4.** QuikChem Method 10-204-00-1-A, Determination of Cyanide in Waters.
- **15.5.** TestAmerica Edison Document No. ED-QA-LQM, *Laboratory Quality Manual*, most current revision.
- **15.6.** <u>TestAmerica Edison SOP ED-GEN-022</u>, *Training*, most current revision.
- **15.7.** <u>TestAmerica Edison SOP No. ED-SPM-008</u>, *Laboratory Waste Disposal Procedures*, current revision.
- **15.8.** <u>TestAmerica Edison Work Instruction # EDS-WI-008</u>, Wetchem Data Review Checklist, most current revision.

#### 16.0. <u>Method Modifications:</u>

ltem	Method no.	Modification
1	335.4	Sec 4.3.1: This SOP referenced method 335.4 (not SW846) when
		treating samples suspected to contain chlorine and nitrates/nitrites.

2	QuikChem Method 10-204- 001-41-A	Sec 7.1.8.3: Preparation procedure for the Phosphate buffer follows Quikchem method 10-204-001-41-1 (not Method 335.4).
3	SM 4500 CN <sup>⊤</sup> C	Sec 10.1.3.10: Sulfamic acid solution is added to all samples during distillation. Addition of Sulfamic acid is a procedure described in Standard methods 18 <sup>th</sup> ed, Method 4500 CN <sup>-</sup> C. Methods SW846 9012B and EPA 335.4 require the addition of Sulfamic acid for samples which are known to contain NO3 and/or NO2.
4	SW846 9012A/9012B	Throughout (as applicable): revised to include the preparation and analysis of wipe samples.

#### 17.0. Attachments

N/A

#### 18.0. <u>Revision History</u>

- Revision 5, dated 22 November 2010
  - Sec. 1.1.1: Added wipes to list of applicable matrices.
  - Sec. 1.1.2: Added reporting limit for wipes.
  - Sec. 6.2: Added specimen cups, transfer pipettes, graduated cylinder and micro-burets to the list of supplies.
  - Sec. 7.1.4: Changed the amount of acetic acid added from "approximately 100 ml" to "approximately 10 drops" to reflect actual laboratory practices.
  - Sec. 7.1.6.2: Clarified reagent prep
  - Sec. 8.0: Added wipes to section.
  - Sec. 9.1: Added sample QC requirements for wipe batches.
  - Sec. 9.1.1: Included the prep of the method blank for wipe batches
  - Sec. 9.1.2: Revised the prep of the LLCS and HLCS to reflect actual laboratory practices. Also, included prep of the LCS/LCSD for wipe batches.
  - Sec. 9.1.3: Revised MS/MSD prep for aqueous and soil.
  - Sec. 9.2.2: Deleted text "If the CCV is not within the control limits, a second analysis should be performed."
  - Sec 9.2.3 & 9.2.4: Clarified the type of solution utilized for the ICB and CCB.
  - Sec. 10.1.3.1: Revised step of the distillation procedure to reflect actual laboratory practices.
  - Sec. 10.2.2.1: Revised calibration standards prep Table.
  - Sec. 10.3.2: Added comment on how to set up the analytical sequence for wipe samples.
  - Sec. 11.5: Included calculation for wipe samples.
  - Sec. 11.6: Added steps to import data to TALS.

- Section 16: Added method revision for addition of wipe matrix to SW846 9012A/9012B.
- Revision 4, dated 28 September 2009
  - Revised SOP format in accordance with TestAmerica Corporate Quality SOP No. CW-QS-002 (Rev 0) Writing a Standard Operating Procedure (SOP).
  - Incorporated SOP ED-WET-007 (Analysis of Total Cyanide in Drinking water-Automated, Method 335.4) into this SOP; retired SOP ED-WET-007 at the effective date of this SOP.
  - Sec. 7.2.2: Added information for the standardization of the Cyanide Stock. Replace frequency of the standardization from monthly to weekly to comply with Standard methods.
  - Sec 7.2.3 7.2.6: Added standards and preparation instructions.
  - Sec 9.1.3: Clarified % recovery limits for water and solid samples.
  - Sec 9.2.1 & 9.2.2: Clarified corrective actions to be taken if QCS/ICV and IPC/CCV results are outside the acceptable limits.
  - Sec 10.3.2: Revised sample analytical sequence to comply with method 335.4.
  - Sec. 11.5: Revised new data reduction procedures in accordance with new TALS.
  - Sec. 15: Replace reference 335.2 and 335.3 with the EPA 335.4 reference; added applicable reference.
  - Sec 16: Added Method modification to specify actual source of reagent information.
- Revision 3b, dated 13 July 2009
  - Sec 2.1: .For each batch of ten samples or less, a matrix spike and matrix spike duplicate will be distilled and analyzed. (Replaced 20 samples to 10 samples per batch).
  - Sec 9.5.1.3 & 9.5.2.2.: Revised Correlation coefficient from 0.997 to 0.995 as per corporate SOP on Calibration curves.
  - Sec 10.3: Up to 10 samples can be put in a batch. .Revised number of samples in a batch from 20 to 10 samples.
- Revision 3a, dated 13 March 2009
  - Sec 1.1: Delete Methods 335.1/.3; Add Method Lachat 10-204-00-1-A (Methods deleted in response to MUR).
  - Sec 3.6: Delete text: If this occurs, a Dow Corning 544 can be added to prevent the foam from collecting in the condenser. Replace the above procedure with: *If this* occurs, samples can be acidified with acetic acid (1.6M) to pH 6.0-7.0.
  - Sec 5.2: Add: Solution is stable for 6 months or when QC check is outside the acceptable limits, whichever comes first, store at 4°C.

- Sec 5.4: Acetate Buffer-Dissolve 82.0g of NaC2H3O2 x 3 H2O in 100ml DI water. Add glacial acetic acid drop by drop until the pH is 4.5. This solution is stable for 6 months, store at 4°C. (Changed amount of acetic acid added to reflect actual laboratory practices).
- Sec 5.6: Remove: Dow Corning 544 antifoam agent. Lab does not use this reagent, samples will be acidified with acetic acid.
- Sec. 5.7.1, 5.8.2, 5.8.3, 5.8.4: Add: Solution is stable for 6 months, store at room temperature.
- Sec 5.7.2: 0.25M NaOH. Add: Dissolve 10.0g NaOH into 1L deionized water. Solution is stable for 6 months, store at room temperature.
- Sec. 5.9.1 & 5.9.2: Add: Store at room temperature.
- Sec. 5.9.3: Phosphate buffer: In a 1L volumetric flask, dissolve 97g of potassium phosphate, monobasic, anhydrous (KH2PO4) in approximately 800ml of deionized water. Dilute to the mark. Solution is stable for one month. Store at room temperature. (Changed reagent preparation to comply with the Lachat method).
- Sec 6.3: Add: Laboratory Control Sample (LCS): Prepare a 5ppm and 10ppm CN standard by diluting the 1000ppm Primary CN stock. Prepare fresh daily.
  - LLCS: Take 0.25ml of the 5ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.025ppm.
  - HLCS: Take 1.0ml of the 10ppm CN standard and bring up to a 50ml final volume with 0.25N NaOH. The true value is 0.2ppm
- Sec 9.10: Delete text: "Free Cyanide is estimated using the weak acid dissociable procedure following the colorimetric procedure without distillation. For reference refer to SM 4500-CN-A." Revise the above text to: "Free Cyanide will be treated following the procedure for the Cyanide Amenable to chlorination and therefore must be distilled." See Section 9.2
- Sec. 10.7: MS and MSD Recovery: Delete: The recovery must be within laboratory generated limits. Revise the above text to: *The recovery must be within 90-110%* (Revise MS/MSD recovery to comply with method 335.4).
- o Sec. 15.1: References: Replace 335.2 & 335.3 reference with EPA 335.4 reference

# **ARCADIS**

# Attachment C-5

Alpha Analytical, Inc. Quality Systems Manual and SOP

# **Quality Systems Manual**

# Alpha Analytical, Inc.

D/B/A

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#### 1 Mission Statement

The mission of Alpha Analytical is quite simply to provide our clients with the greatest value in analytical service available. For the 'greatest value' is not only found in the data that is delivered, it is also found in the services provided.

- Data must be of the highest integrity, accuracy and precision.
- Consultation and educational services must be provided to support the customer in establishing data quality objectives and interpretation of the final data package.
- Support services such as sample containers, courier service and figure on data deliverables must be available to the client.

Alpha's mission continues with an established commitment to our community and environment. We must ensure that we do not produce any additional communiction to our environment or harm our neighbors and community in any way.

The value of Alpha's product is in the honest, and so grity with which each chemist, courier, login staff member, or office staff member, performs their tasks. The client or employee must always feel satisfied that they received the greatest value in their lab experience at Alpha.

Alpha Analytical Labs will vigorously pusue its mission into the next millennium.

Mark Woelfel

President

Document No.: CQSM/01 Issue Date: Jul 22, 2009 Issue No: 2 Effective Date: Jul 31, 2009 Page 1-2 of 2 Uncontrolled Document 1.1 Signature Page Copy No. \_\_\_ 1.2 **Management Authorization** President Signature: Date: 7-22-09 Name: Mark Woelfel Quality Assurance Officer Signature: 2.09 'ete: Name: James Todaro Laboratory Director / Technical Director (Westboro) Signature: 4 late: Name: Christøpher Wakefield Laboratory Director / Technical Direct. (Mans. 3ld) 7/22/09 Signature: 5 Date: Name: Leonard Pitts **Technical Director** 7-22-0 Signature Date: Name: An ' Rezendes The above signed understand and acknowledge that Alpha Analytical is required to be continually

in compliance with the National Environmental Laboratory Accreditation Conference (NELAC) standards

ISSUE AMENDMENTS

None

Quality Systems Manual

Alpha Analytical

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#### 3 Introduction

The Quality Systems Manual (QSM) of Alpha Analytical describes the quality program in use at the laboratory for both Westboro and Mansfield facilities. This Quality Systems Manual provides employees, clients and accrediting agencies with the necessary information to become familiar with how the quality system operates within Alpha Analytical. The quality program includes quality assurance, quality control, and the laboratory systems including feedback mechanisms for the automated continuous improvement of the laboratory operations to meet client needs.

Implementation of the laboratory operations is by documenting procedures, training personnel and reviewing operations for improvement. Written procedures are maintained as Standard Operating Procedures (SOPs). The SOPs are available to the staff as a uncontrolled, electronic, secure copy. The provisions of the QSM are binding on all temporary and permanent personnel assigned responsibilities. All laboratory personnel must adhere strictly o the SM and SOPs.

All policies and procedures have been structured in accordance wit', the National Environmental Laboratory Accreditation Conference (NELAC) standards ap 'cable EPA requirements and applicable Department of Defense (DOD) Quality System. Ma. 1ai, ...ev 4.1 standards.

Fifteen (15) sections comprise the QSM. Related chain, dow mentation including the listing of SOPs, forms, floor plan, equipment, personnel and labor bory qualifications are available. The QSM sections provide overview descriptions of pages. As policies, services and operations.

#### 3.1 Scope

The QSM describes the requirement of the Laboratory to demonstrate competency in the operations for performing environment tests for inorganic, organic, air and microbiological testing. The basis for the environment tests is the methods found in documents published by the United States Environmental. Protection Agency (EPA), ASTM, AOAC, APHA/AWWA/WEF, Standard Methods, DOD-C SM 4.1, and other procedures and techniques supplied by clients.

The QSM includes equipments and information for assessing competence and determining compliance by the labore bry to the quality system. When more stringent standards or requirements are bolded in a mandated test method, by regulation, or specified in a project plan the labore tory dei postrates achievement of the client specified requirements through its documente, processes.

The QSM is for use by Alpha Analytical for developing and implementing the quality system. Accrediting authorities and clients use the QSM for assessing the competence of Alpha Analytical. Alpha Analytical is committed to continually improving the quality system. Meeting customer needs, operating within regulatory requirements and adhering to Alpha's Data Integrity and Ethics policy are several of the mechanism used to continually improve the quality system.

#### 3.2 Policy Statement

This Quality Systems Manual summarizes the policies, responsibilities and operational procedures associated with Alpha Analytical. This manual applies to all associates of the laboratory and is intended for use in the on-going operations at Alpha Analytical. Specific protocols for sample handling and storage, chain-of-custody, laboratory analyses, data reduction, corrective action, and reporting are described. All policies and procedures have been structured in accordance with the National Environmental Laboratory Accreditation Conference (NELAC) standards, applicable EPA requirements, regulations, guidance, and technical standards and DOD QSM 4.1 standards. This Quality Systems Manual, laboratory Standard Operating

Procedures (SOPs), and related documentation describe the quality systems, policies and procedures for Alpha Analytical.

Alpha Analytical performs chemical analyses for inorganic and organic constituents in water, seawater, soil, sediment, oil, tissue and air matrices. Alpha Analytical's goal is to produce data that is scientifically valid, technically defensible, and of known and documented quality in accordance with standards developed by NELAC and any applicable state or EPA regulations or requirements. It is the commitment of the President, Operation Director, Laboratory/Technical Director and Quality Assurance Officer to work towards continuous improvement of the operation, and towards meeting our client's needs, requirements, and intended data usage. This continued commitment is built into every activity of the laboratory. It is the responsibility of Senior Management and the Department Managers to ensure that all associates familiarize themselves with, and comply at all times with, the quality systems, procedures and policies set forth in this manual, laboratory SOPs, and related documentation.

Alpha Analytical analyzes Proficiency Test (PT) samples, in accorda ce w. b NELAC and other regulatory programs, from a National Institute of Standards and Techr. logy (NIST)-approved PT provider for the analytes established by EPA for water samples, ind for other analytes and matrices. The specific analytes and matrices analyzed a bare or the current scope of the laboratory services as documented in the laboratory SO, sail 'static artifications.

The technical and service requirements of all rections to provide analyses are thoroughly evaluated before commitments are made to accept he work. This includes a review of facilities and instrumentation, staffing, and any special QC or r porting requirements to ensure that analyses can be performed correctly and within the expected schedule. All measurements are made using published reference methods or methods developed by Alpha Analytical. Competence with all methods is demonstined correcting to the procedure described in SOP/ 08-12 prior to use.

Alpha Analytical has developed a projective program for prevention and detection of improper, unethical or illegal actions cont, on ints of this program include: internal proficiency testing (single and/or double blin 1); ele tronic data audits and post-analysis data review by the QA Officer; a program to in provide moloyee vigilance and co-monitoring; and Ethics Training program identifying appropriate and inappropriate laboratory practices, instrument manipulation practices and consequentes. Additionally, all associates are required to sign the Alpha Analytical *Ethics Agreement* form u, on commencement of employment and each year following. This form clearly outlines the possible consequences of unethical or improper behavior, or data misrepresentation.

It is the policy of the laboratory to discourage and reject all influence or inducements (whether commercial, financial or personal) offered either by customers or suppliers, which might adversely affect results or otherwise compromise the judgment or impartiality of the staff. It is the responsibility of the Operations Director and Laboratory/Technical Director to inform customers and suppliers of this policy when necessary.

In the event that any such influences or inducements are encountered, the staff is instructed to inform management immediately. It is the responsibility of the Operations Director and the Laboratory/Technical Director to take appropriate action to prevent recurrence.

# 3.3 References

An electronic register of external documents or books is available on the company intranet for staff to determine the latest edition or version of the reference methods, regulations or national standards. The Quality Assurance Department maintains the register. Management purchases automated update services, where available, to provide the laboratory with the latest hardcopy edition, where electronic means is not available.

#### 3.4 Definitions

Appendix A lists the definitions as adopted by the lab, ato, The definitions are from the standard approved in June, 2005, by the Nation a Thui, nmental Laboratory Accreditation Conference (NELAC). The definitions in Appendix A are up lated, as necessary, after publication of the NELAC adopted Glossary.

#### 4 Organization and Management

#### 4.1 Legal Definition of Laboratory

Alpha Analytical is a full service analytical laboratory. Testing services include Drinking Water, Waste Water, Ground Water, Waste material and Air. Alpha Analytical is a privately held corporation incorporated in the state of Massachusetts. Alpha Analytical, Inc. does business as (D/B/A) Alpha Analytical.

Alpha Analytical has been in business since 1985. The types of businesses served include:

- Consulting firms,
- Engineering firms,
- Waste Management Companies,
- Industrial sites,
- Municipal agencies and
- Other commercial businesses.

#### 4.2 Organization

The laboratory operates a quality system a groath to management in order to produce data of known quality. The laboratory organization provides effective communication and lines of authority to produce analytical data rise, og client specifications. The organizational design provides open communication while ensuring that pressures and day to day operating circumstances do not compromise for integration arity of the reporting of the final data.

The President is responsible for direct ig all areas of the company. The following job functions report to the President:

- Operation J. 'and J
- Quality, asural, e Officer
- Clien, Perv. es Manager
- Marketir J / Business Development / Sales
- F. ancial Services
- Human Resources

The Operations Manager is responsible for directing all laboratory operational areas of the company. The following job functions report to the Operations Manager:

- Laboratory/ Technical Director(s)
- Department Managers

The Laboratory/Technical Director(s) is responsible for the laboratory data generated by the organics testing, inorganics testing and metals testing areas and the Air Technical Director is responsible for laboratory data generated by air analyses.

The Departmental Managers (Supervisors) have the following responsibilities:

- The organics managers direct personnel in the organics extraction and instrumental laboratories.
- The wet chemistry manager directs personnel and team leaders in the wet chemistry and/or microbiological testing areas.
- The metals manager directs personnel and team leaders in the metals sample preparation and instrumental laboratories.

The Quality Assurance Officer is a member of the staff reports directly the President and has defined responsibility and authority for ensuring that the quality system, is implemented and adhered to at all times. The Quality Assurance (QA) Officer is responsible for interacting and communicating certification requirements, implementing the Castlin, Sistems Manual and reporting to the Laboratory Director and Senior Management files, at is of the quality program. The QAO oversees the Quality Systems Specialists and is responsible or oversight and/or review of quality control data and function independently from laboratory operations.

The Client Services Manager is responsible for client intelactions, project coordination and laboratory personnel notification of project required ents. Also the Client Services Manager is responsible for the areas of sample container steps, the and transportation of containers and samples to and from the laboratory.

The Marketing, Business Development an, Sa, personnel are responsible for increasing the volume of work from current clients ind acting new clients to the base business of Alpha Analytical. The Marketing and Business Development personnel review all new work with the Laboratory Director, Operations Manager, President and/or Quality Assurance Officer before contractual commitment.

The Controller is responsite a for r aintaining and reporting on the financial status of the company. The Controller directromanc. "Dersonnel on proper accounting procedures and maintaining the list of approver' suppriers a. I subcontractors. The Controller reports directly to the President.

The Human Rescirce Director is responsible for personnel recruitment, hiring, performance reviews.

Personnel job descriptions define the operational function duties and responsibilities. Administration and Laboratory personnel assignments may include cross-functional training and work performance in multiple areas of the operations. Multiple function training ensures laboratory back up personnel during peak work loads.

During the absence of any staff member, assignment of alternative personnel occurs by memo or e-mail. The Manager or Supervisor authorizes the assignment. The naming of alternative personnel assures the continuing performance of critical tasks during the primary person's absence and ensures that lines of communication remain open for continued decision making. The deputy for the Laboratory Director is the Quality Assurance (QA) Officer. The deputies for the Quality Assurance (QA) Officer are the Quality Systems Specialists.

For the purposes of NELAC Accreditation and DOD QSM 4.1, the Lead Laboratory Technical Director is the Laboratory Director. The deputies for the Lead Technical Director are the Quality Assurance (QA) Officer, and the Departmental Managers. The Laboratory/Technical Director meets the requirements specified in the Section 4.1.1.1 of the NELAC standards. If the Technical Director is absent for a period of time exceeding 15 consecutive calendar days, a full-time staff

member meeting the qualifications of Technical Director will be designated to temporarily perform this function. The primary Accrediting Body shall be notified in writing if the Technical Director's absence exceeds 65 consecutive calendar days.

#### 4.3 Business Practices

Alpha maintains certification for the programs and analytes required by regulatory programs. The listing of qualifications from the various certifications, registrations and accreditation programs are available upon request. Alpha Analytical operates Monday to Friday from 7:30 a.m. to 5:30 p.m. Management prepares and posts the holiday schedule for the year indicating closed operations. Sample delivery occurs during normal operating hours unless arranged in advance.

Alpha's reputation depends upon timely reporting and quality data. The standard turnaround time for engineering and consulting firms is five business days from time of ample receipt. Standard turnaround for all other clients is ten business days from time of scale, receipt. The time of sample receipt is when the verification of the chain of custody and samples meets the laboratory sample acceptance policy. Laboratory management must approve any meet all arrangements for rush or expedited turnaround time. The basis for data quality (spelids on client, regulation and method performance criteria. Accuracy, precision, sensitivit, and comp rability are expressions of method performance criteria.

All work is performed in the strictest confidence. New and contract employees must review corporate policy and practice requirements for projecting client confidentiality and proprietary rights. The review occurs during orientation and thic, trailing. It is the policy of the laboratory to release data to the client authorized conte. The sonnel assigned the duties of interacting with clients review project files and discuss data relief only to the project. Personnel whose duties do not include routine client contact must another with the client service manager before discussing data with regulators or third parties.

## 5 Quality System

Establishment, Audits, Essential Quality Controls and Data Verification

### 5.1 Establishment

The Mission Statement presents the policy and objectives for Alpha Analytical. The Quality Systems Manual provides the framework for the processes and operations to implement the Mission. The Quality Systems Manual and documentation controlled by the laboratory system detail the management authorized operations for achieving the objectives of the company.

The laboratory operates a quality system approach to management in rder to produce data of known quality. Alpha Analytical is a full service laboratory designed to provide its clients with accurate, precise and reliable data within the best turn-around time and at the most reasonable prices. Alpha employs chemists of the highest training, ethics and the liber in the field of analytical chemistry. This and state-of-the-art instrumentation and autor ation of the insure data of known and documented quality.

# 5.2 Quality Systems Manual

The QA Officer is responsible for the publication and distril ution of the Quality Systems Manual. Management reviews and authorizes the nanual Implementation of major changes in the quality system occurs after revision of the appropriate Quality Systems Manual section and authorization by management.

The authorization signatures found or, the signature page of the manual signify management review and approval of the Qualit, S, ter. 3 Manual. The Signature section must be kept current and reflect any organizational changes ffecting the authorizing positions. Updates of this manual occur at any time throughor. The rear The issue number and date are changed to denote the latest revision date. The revision date for the signature section must be the most recent, indicating that all revisions. The revision management review.

Document cor. of procedures (SOP/08-01 Westboro and SOP/G-016 Mansfield) apply to the distribution of the Dual. / Systems Manual. Distribution of controlled copies of the manual is only to an individual within the laboratory. Persons or organizations outside of Alpha Analytical may receive uncontrolled copies. Copies are distinctly marked "Uncontrolled Documents". A distribution list maintained for all controlled copies of the Quality Systems Manual. All parties listed on the controlled distribution list receive document updates. Copies marked as uncontrolled copies are not subject to updates.

# 5.3 Audits

Laboratory audits, both internal and external, review and examine the operations performed in the laboratory. Internal audits are condcted by qualified QA Specialist and external audits are reviews by external organizations to evaluate the ability of the laboratory to meet regulatory or project requirements.

A QA designee schedules internal process audits to ensure the completion of the annual audit of each operational area. The process audits are a more detailed review of the operations. Personnel from areas other than the one audited perform process audits.

The internal system audit is a review of the implementation of the documented quality system. The system audit includes sample tracking from receipt to disposal, a data audit of a completed report, and all operations not audited during the process audit.

The purpose of the internal system audit is:

- Verification that adequate written instructions are available for use;
- Analytical practices performed in the laboratory are consistent with SOPs;
- The quality control practices are applied during production;
- Corrective actions are applied as necessary;
- Deviations from approved protocols are occurring only with proper authorization and documentation;
- Reported data is correct and acceptable for reporting;
- SOPs, quality records, analytical records, electronic data files a p maintained properly; and
- Personnel training files and records are satisfactory file in the satisfactory file

Before a scheduled audit, the assigned auditor reviews the dist and or the SOP specific to the area. The checklist may be from an external source or papaled by the auditor. The checklist must include all references to the documented or the extern or referenced requirements document. After the audit, the auditor submits  $\epsilon$  summary or notes from the audit to the Laboratory Director or QAO as part of the audit report. The summary identifies discrepancies found during the audit. A copy of the summary form to presented to the person responsible for implementation or resolving the problem. The summary comprise the audit report.

Technical personnel are responsible or the inspection and monitoring of in-process and final data. Personnel independent of the secthal guide the guality system and processes.

Representatives sent by c ents and government or accrediting agencies often perform external audits. These audits are n. st off in announced inspections, but sometimes are not announced. The Quality Assurance Orneer, Laboratory Director or assigned deputy, or appropriate Department M. hage accort panies the external audit team through the laboratory. The auditors receive a brief or rvie, of company objectives, activities, and facilities. Interviews with essential supervise v staff a. I technical staff are arranged, along with retrieval of any documentation pertinent to the audit. Auditors usually provide a report on their findings shortly after the audit. The QA Office regives the audit report and copies are provided to laboratory personnel for review. Corrective actions are identified and distributed to responsible parties for implementation in response to any cited deficiencies.

### 5.4 Audit Review

Management reviews internal and external audit reports to evaluate system effectiveness at the annual management review meeting. Tracking of the audit findings occurs through the nonconformance action process. The management and staff work together to establish a time line for resolving the audit findings. The Quality Assurance team tracks the time line and reports to the Laboratory Director on any outstanding audit findings.

### 5.5 Performance Audits

Alpha Analytical participates in inter-laboratory comparisons and proficiency test programs required by clients and certifying agencies. The performance audits provide information on the data comparability of results generated by the laboratory. Test samples received by the

laboratory are handled following routine laboratory procedures. Proficiency test samples are unpacked, checked against the packing slip and examined for damage. Reporting requirements and deviations to routine practices are noted as would be required for any project.

Analysts demonstrate proficiency by analyzing either an external proficiency test sample, an internally prepared blind test sample or Initial Demonstration of Capability (IDC) before independent operation of a test method and at least once per year per analyst. The results of performance audits serve several purposes. The QA Officer may use performance audits for evaluating analyst proficiency, laboratory performance in a specified area to facilitate laboratory improvement efforts, and/or to provide information to an accrediting agency on correction of past-performance of an external performance audit.

# 5.6 Corrective Actions/Preventative Actions (CAPA)

The corrective action process at Alpha Analytical is detailed in SOP/0P  $_{\rm OP}$ . The corrective action program at Alpha Analytical uses the Nonconformance Report for to a sument and follow through the corrective action/preventative action process for three main area: nonconformance's within the laboratory, client complaints and failed PT studies. The mechanism for recording, reviewing and acting upon all quality problems is self-explenated as the form is completed. The process ensures continuous improvement of company performance by preventing the recurrence of quality problems.

Clients will be notified within 5 days of iny quartion(s) regarding validity of results.

# 5.7 Managerial Review

The management review clours of least once per year as part of the strategic planning process. Documentation of the mailagement review meeting is by recording the meeting minutes and listing the attendees. The focus of the quality management review is the frequency of the type of nonconformance, claure status, audit progress and other quality assurance actions. Meetings include discussio, and progress on quality system initiatives since the last meeting.

Prior to the peeting an agenda is distributed to all personnel expected to be in attendance. The meeting is charged by the Quality Assurance Officer. Minutes are taken and distributed at the conclusion of the meeting by a QA designee. If action is necessary on any issue, a Summary Report is generated and distributed to responsible parties for implementation. Actions are monitored by the QAO or designee until completion.

# 5.8 Essential Quality Control Procedures

The following general quality control principles apply to all tests. The manner implemented is dependent on the type of test performed. The laboratory SOP presents the specific quality control checks undertaken to ensure precision, accuracy and sensitivity of each test method.

Alpha Analytical uses quality control samples to evaluate the following:

- 1. Adequate positive and negative controls to monitor blanks, spikes, reference toxicants, zero blanks;
- Adequate tests to define the variability and/or reproducibility of laboratory results;

- Measures to ensure the accuracy of the test data including sufficient calibration and/or continuing calibrations, use of certified reference materials, proficiency test samples;
- 4. Measures to evaluate test performance, such as method detection limits and quantitation limits or range of applicability such as linearity;
- 5. Selection of appropriate formulae to reduce raw data to final results such as linear regression, internal standards, or statistical packages;
- 6. Selection and use of reagents and standards of appropriate quality;
- 7. Measures to assure the selectivity of the test for its intended purpose;
- 8. Measures to assure constant and consistent test conditions for the method such as temperature, humidity, light, or specific instrument conditions.

All quality control measures are assessed and evaluated on an corgon a basis, and quality control acceptance limits are used to determine the usability of the cata. Control charts and/or calculated control limits monitor the long-term method performance of the cata. Control charts and/or water matrices. Routine evaluation and reporting of the control chart performance provides supervisors and management with additional peformance measures to ensure data comparability. Control limits are recalculated when trends are conserved.

Where no reference method or regulatory crieria xis., the laboratory specifies the acceptance/rejection criteria in the SOP.. The test S DP spicifies the QC samples performed per batch of samples. The quality control samples fre ca. United into the following, as appropriate to the method

- Method Blank
- Laboratory Duplicate
- Laboratory Cont الا Sa ple (LCS)
- Laboratory Cont. I San ple Duplicate (LCSD)
- Matrix Spi' a (MS)
- Matrix Spik Dur cate (MSD)

The frequency is dependent on the reference method and test protocol. The following is the default requirement of quality control checks in lieu of any other guidance. The frequency for each quality control sance is generally one (1) per every 20 samples.

# 5.9 Data Reduction

After completion of the test procedure, the data reduction process begins.

Chromatography data may require the manual integration of peak areas or heights before reporting of results. The analyst must perform manual integration when software does not properly integrate or identify the peak. Manual integration must not occur for the purpose of achieving acceptable quality control or calibration. The analyst and reviewer sign and date the hardcopy of all manual integration. The analyst notes the rationale for performing the manual integration on the hardcopy printout and ensures the "TIC" marks from the software represent the integration area used for reporting the results. The analyst must minimize and avoid manual integration. The establishment of the proper integration parameters in the software reduces the number of manual integration occurrences.

The SOP for each test presents the formulas used for the specific test method. The formulas for the data calculations used throughout the laboratory are the following:

% Recovery (LCS)  $\frac{MV}{TV} * 100 = \% R_{LCS}$ *MV* = Measured Value *TV* = True Value where: % Recovery (MS or MSD)  $\frac{MV - SV}{TV} * 100 = \% R_{MS}$ ΜV = Measured \ alu where: TV = True 'alue sv = Amou... hund in sample Average ( $\overline{X}$ )  $\sum_{i=1}^{n} X_{i} / n$  $\begin{array}{c} \overline{\mathbf{v}} \\ \mathbf{x} \\ = \\ = \end{array}$ Average of all values Result of each measurement where: Number of values Relative Percer Difference (% RPD)  $\frac{R_1 - R_2}{(R_1 + R_2)/2} *100 = \% RPD$  $R_1$  = Larger of two observed values  $R_2$  = Smaller of two observed values where: % Difference (%D)  $\frac{X - \overline{X}}{\overline{X}} * 100 = \%D$  $\overline{X}$  = Average of all values X = Result of measurement where:

Standard Deviation of the sample  $(S_x)$ 

$$\sqrt{\frac{\sum (x - \overline{x})}{n-1}} = S_x$$
ere:  $\overline{X} = Average of all values$ 

$$X = Result of each measurement$$

$$n = Number of values$$

Relative Standard Deviation (%RSD)

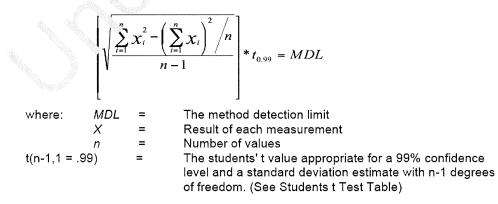
wh

$$\frac{S_x}{\overline{X}} * 100 = \% RSD$$
where:  $\overline{X} = Av_{t-1}ge c^{t} all values$ 
Sx = Stanc rd D viation (n - 1)

Range of Logs (for microbiological e umeration analysis)

10% of routine samples are . nalyze I in duplicate and the range of logs is determined.

MDL (See 40CFR Pan 136 fc details)



Reporting Limit (RL)

Lowest calibration standard or greater At least 3 times the calculated MDL

Reportable Detection Limit (RDL)

Lowest calibration standard or greater adjusted for sample/matrix

Control Limits		
	Upper Control Limit:	$\overline{X}$ + 3 * $S_x$ = UCL
	Lower Control Limit:	$\overline{X} - 3 * S_x = LCL$
Warning Limits		
Ū	Upper Warning Limit:	$\overline{X} + 2 * S_x = UWL$
	Lower Warning Limit:	$\overline{X} - 2 * S_x = \mathcal{O}W$

Method of Standard Additions (MSA): (See EPA 7000A, r de sils)

The simplest version of this technique is the  $\sin \sqrt{3}$ -addition method, in which two identical aliquots of the sample solution. ach o volume Vx, are taken. To the first (labeled A) is added a known volume V: of  $\sqrt{3}$  star dard analyte solution of concentration Cs. To the second aliquot (labeled  $\sqrt{3}$ ) is dded the same volume Vs of the solvent. The analytical signals of A and B are me, sure and corrected for non-analyte signals. The unknown sample concentration  $\sqrt{2}$  call tated:

 $C_{x} = \frac{SB V_{S} C_{s}}{(SA - SB) V_{x}}$ 

where SA and SB re the analytical signals (corrected for the blank) of solutions A and B, respectively. Young Theorem is chosen so that SA is roughly twice SB on the average, avoiding excess a vition of the sample. If a separation or concentration step is used, the addition are estimate first and carried through the entire procedure.

In, roved re ults can be obtained by employing a series of standard additions. To equal volu, as of the sample are added a series of standard solutions containing different known quantities of the analyte, and all solutions are diluted to the same final volume.

For example, addition 1 should be prepared so that the resulting concentration is approximately 50 percent of the expected absorbance from the endogenous analyte in the sample. Additions 2 and 3 should be prepared so that the concentrations are approximately 100 and 150 percent of the expected endogenous sample absorbance.

The absorbance of each solution is determined and then plotted on the vertical axis of a graph, with the concentrations of the known standards plotted on the horizontal axis. When the resulting line is extrapolated to zero absorbance, the point of interception of the abscissa is the endogenous concentration of the analyte in the sample. The abscissa on the left of the ordinate is scaled the same as on the right side, but in the opposite direction from the ordinate. A linear regression program may be used to obtain the intercept concentration.

# 5.10 Document Control

The Document Control Procedures (Westboro SOP/08-01 and Mansfield SOP/G016) describe the process for controlled and uncontrolled documents. The use of the issue number allows for the retention of a previous document for historical information purposes.

Every document is assigned a unique identification number, which is place. \* on each page of the document. A master list of documents includes the unique identification. E ch controlled copy includes the issue number, issue date, effective date and page number.

Full document control includes the status of each chaumint: active, inactive or superceded/archived. Inactive documents are procedure, not surreinly requested, but may be in the future. Archived documents are procedures replaced with a later revision. Authorized personnel must review and approve each document and my absequent revisions before use in the laboratory. Personnel authorized to review and approve a document have access to all necessary information on which to base their review and exproval. The amendment section of the signature page of any SOP includes a briet document is status of the nature of the document change.

Standard Operating Procedures (SOP', e in. ' uctions for repetitive or standard operations performed by the laboratory. The SO, autho. is the person familiar with the topic. The standard format for writing SOPs is set-up and the plate for administration and technical SOPs. Each SOP is peer reviewed, authorized by mana encent, and the Laboratory Director or QAO before final distribution and implementatio. Pe sonnel acknowledge approved documents as read, understood and agreed to hypersc. In through signed attestation forms or training records.

SOPs must receive  $\epsilon$  wate and input by laboratory supervisors and key technical personnel. The content of eac's SOF must conform to applicable requirements of analytical methods and certification age dies. Within these constraints, the content of a SOP meets the needs of a particular area of the laboratory. A new or revised SOP is needed when regulatory programs update or the discort of the existing method is extended or when activities are being performed without adequate documentation.

Updating, modifying and changing SOPs, forms and the contents of this QSM are prompt and part of the routine practices. The prompt modification of these documents ensures the documents reflect the current practices and operations of the laboratory. Implementation of modifications required before issue of an updated SOP is authorized by the Departmental Manager on the SOP Review Form. The record of the SOP change authorization by management, QA and other analysts is placed on the nonconformance form. A copy of the approved nonconformance form is retained with the SOP. During annual review of a document, (including but not limited to: SOPs, Ethics Policy, Quality Systems Manual), requested changes are reviewed and the document reissued using the information from the nonconformance forms.

The laboratory maintains control over the possession and distribution of all documents that directly affect the quality of data. This includes, but is not limited to, documents such as the Quality Systems Manual, Standard Operating Procedures, client instructions, Laboratory Work Instructions, data sheets, check lists and forms.

# 5.11 Detection Limits

Method Detection Limits (MDLs) are determined for all analytes as specified in the NELAC and other standards. MDLs are determined for all new instrumentation, whenever there is a change in the test method or instrumentation that affects performance or sensitivity of the analysis. From these, detection limits, practical quantitation limits (PQLs), or Reporting Limits (RLs), are established. The PQL is the minimum concentration of an analyte that can be identified and quantified within specified limits of precision and bias during routine and analytical operating conditions.

Laboratory reporting limits lie within the calibration range, at or above the PQL. For methods that require only one standard, the reporting limit is no lower than the low-level check standard, which is designed to verify the integrity of the curve at lower levels. If reporting in. its are required below the lower level of the calibration curve, PQL, or low-level check stan ard, r. thod modifications are required. Refer to MDL/LOD/LOQ SOP/08-05.

# 5.12 LOD/LOQ Studies

### A. LOD (Limit of Detection) Verification

- 1. LOD (Limit of Detection) verification is require, annually for each target analyte in which test results are to be reported below the lo rest calibration standard ("J" values) for each instrument, matrix and prep proced re.
  - a. Quarterly LOD Ver ication. is required for DOD projects.
- 2. All sample-processing steps of the nalytical method shall be included in the determination of the LOD.
- 3. The validity of the I OL shall be confirmed by <u>qualitative</u> identification of the analyte(s) in a QC sample in erich value system matrix containing the analyte at no more than 2-3X the LOD for s. all halve ests and 1-4X the LOD for multiple analyte tests. This verification must be per, rme on every instrument that is to be used for analysis of samples and reporting of date
- 4. An LOD s. ... s not required for any component for which spiking solutions or quality control samples are not available such as temperature. Where an LOD study is not performed, the laboratory may not report a value below the limit of quantitation.

### B. LOQ (Limit of Quantitation) Verification

- LOQ (Limit of Quantitation) verification is required annually for each target analyte that is not reported below the lowest calibration standard for each matrix and prep procedure. LOQ is not required if an annual LOD verification is performed.
- 2. The validity of the LOQ shall be confirmed by successful analysis of a QC sample containing the analytes of concern in each quality system matrix 1-2 times the claimed LOQ. A successful analysis is one where the recovery of each analyte is within the established test method acceptance criteria for accuracy.

The LOQ study is not required for any component or property for which spiking solutions or quality control samples are not commercially available or otherwise inappropriate (e.g., pH).

The LOQ acceptance criteria is based on the established acceptance criteria for Laboratory Control Samples.

Refer to MDL/LOD/LOQ SOP/08-05.

# 5.13 Range of Logs – Precision of Quantitative Methods - Microbiology

- A. Precision of duplicate analyses is calculated for samples examined by enumerative microbiological methods according to the following procedure:
  - a. Perform duplicate analyses on first 15 positive samples.
  - b. Record duplicate analyses as D1 and D2 and calculate the logarium of each result.
  - c. If either of a set of duplicate results is <1, add 1 to oth verues before calculating the logarithms.
  - d. Calculate the range (R) for each pair of transformed 'uplicates as the mean of these ranges.

# 6 Personnel

# 6.1 Laboratory Management Responsibilities

Management is responsible for communicating the requirements of the quality system, client specifications and regulatory needs to all personnel. Management job descriptions detail the responsibilities of each position.

H.R. Director has job descriptions for all positions in the laboratory defining the level of qualifications, training, and experience and laboratory skills. During init, ' training, management provides documented operations procedures, observes personnel proformance, and evaluates personnel proficiency. Management documents technical laboratory st. ff's production y initially and on a continuing basis through use of laboratory control samples and proficiency evaluation standards. Management requires successful proficiency are postration before allowing independent production testing.

Management is responsible for verification of proper sample nanagement and all aspects of data reporting. The communication of the operating ractic s of the laboratory is through the document control and attestation process.

# 6.2 Laboratory Staff Requirements

Recruitment is the responsibility of the Dperctions Manager and HR Department, with input from other personnel as required. The Training Program procedure SOP/15-01 details the process for completing requirements and training the cure personnel have adequate skills and competence for the job function.

A job description details the nelessary requirements for each job and includes position title, minimum educational equivaments, skills, responsibilities and reporting relationships and any supervisory responsibility.

Initial training of n v en ployees and contract staff includes laboratory ethics and quality policies, as well as execution of an Ethics Agreement. Any employee found to knowingly violate the Ethics Policy Agreement, eport data values, that are not actual values obtained or improperly manipulated, or mentionally report dates and times of data analyses that are not the actual dates and times of analysis, will lead to disciplinary action, including termination, as outlined in Section K of the Employee Handbook. Each employee must report personally or anomously to the Laboratory Director, QA Officer and/or Ethics Team Member any accidental or suspected intentional reporting of non-authentic data by others for follow up action. The review of the laboratory ethics policy occurs annually with all personnel. The annual review includes annual renewals of the Ethics Agreement.

The Ethics program consists of the following key components:

- Ethics Policy /Agreement (Appendix F)
- Initial and annual ethics training
- Internal audits conducted annually
- Adherence to Manual Integration SOP/08-03

- Ethical or Data Integrity issues reported to Lab Managers, QAO or HR Director
- Anonymous reporting to HR Director
- "No-fault" policy encouraging reporting of incidences without fear of retribution
- Electronic tracking and audit trails through LIMs and instruments enable where available.

## 6.3 Training

The Quality Systems Manual and related documentation is available to all employees. Cross training, supervisory training and other related training takes place on a scheduled and asneeded basis. Training ensures the communication and understandir, of all personnel in the laboratory-documented procedures and practices.

All personnel undertake orientation-training sessions upon initial emplo, men'. Orientation training includes laboratory business practices, employment specifications, Funics Policy, Quality Systems Manual, Chemical Hygiene Plan, and all SOPs required for the jub function.

Managers ensure the training for new employees and review Let continuing training for current employees. Training includes on-site and off-site programs presented by staff members, contractors, equipment manufacturers, and institutions of his her learning.

Training of new personnel to any job ass, nme. ' takes place on-site according to the Training Program procedure. Laboratory personnel new perior m their assigned methods/protocols without supervision only after documentation of accepted proficiency. Training records lists the current training status.

On-the-job training includes demonstration of skills during job performance, initial demonstration of proficiency, and review of SCPs. So jety and health training takes place on an annual basis with careful introduction to the proficiency, and review of SCPs. So jety and health training takes place on an annual basis with careful introduction to the proficiency and review proficiency. So personnel have access to the Chemical Hygiene Plan and Material Safety Data theets. On-site training includes side-by-side hands-on training, formal classroom type instruction of the SOP or a meeting to discuss procedural changes or to address questions related to the 'aboratory operation. All training is documented via the Training Attestation Form why his signed by all in attendance that they understood and will implement what was presented to chem.

Training is a on-gr ing opportunity to evaluate the laboratory operations. The updating of SOPs, Quality System in Annual and other related information documents all changes to the quality system. In all cases, training is documented via the Training Attestation Form.

Off-site training takes place on an as-needed basis. Recommendations and suggestions regarding educational programs come from all levels of staff. It is the employee's responsibility to present a copy of any certificates or attendance information to the HR Director. The information is added to the individual's training record.

### 6.4 Records

The QA Department is responsible for maintaining training records. Attestation forms, certificates, demonstration of capability forms and other records of training are placed in the individual's training file.

The Quality Assurance Officer or designee notifies appropriate personnel when a revision is complete for the controlled version of a document. Laboratory staff must implement the change as of the effective date. The manager of the area determines when a change is significant to require training.

Job descriptions are included in the training record files. The Human Resources Department reviews the job descriptions, resumes and training records to ensure up-to-date information on the job descriptions and resumes. The Human Resources Department and the individual update the resume on an as needed basis. Resumes and/or biosketches are kept on file with the Human Resources Department and the QA Department.

# 7 Physical Facilities – Accommodation and Environment

This laboratory facility has a total area of 25,000 square feet in both the Westboro and Mansfield Facilities

The laboratory functional areas include:

- Administration and offices
- Sample receiving
- Sample management
- Microbiological (Westboro Facility only)
- General analytical chemistry
- Metals sample preparation
- Organic sample preparation
- Metals analysis
- Volatiles gas chromatography (GC)
- Volatiles gas chromatography/mass spectrometi, (GU/MS)
- Volatiles air analysis (Mansfield Facility nly)
- Semivolatiles gas chromatography/mass mect smetry (GC/MS)
- Semivolatiles gas chromatogra, (C<sup>1</sup>)
- Miscellaneous facility mechanical and storage areas.

All chemicals are stored in appropries cabinets and properly disposed of as required. All flammable solvents are stored in Co. A and NFPA approved cabinets. Acids are stored in OSHA acid cabinets. Separate waste  $\epsilon$  eas houses the sample and chemical waste before pickup by a licensed waste hauler.

# 7.1 Environment

Lighting, noise, um, 'ty, heating, ventilation and air conditioning satisfy the needs of the testing performed on the premises. The laboratory building design ensures regulated temperature control for analy, all equipment. Air-handling systems minimize airborne contaminants that may jeopardize scape i legrity or analytical performance.

The analytical instrumentation is in separate rooms from laboratory activities that involve the use of large quantities of organic solvents or inorganic acids. A separate room, in the Westboro facility, provides the facilities for the microbiological testing.

Standards and other materials requiring below 0°C storage temperatures are placed in freezers and separated from samples or potential contaminating materials. Refrigerators provide cooling needs for samples and materials with temperature requirements of below room temperature and greater than freezing. Sample and standard storage areas are monitored and controlled for temperature. Sample storage areas for volatiles are separated from other samples and monitored for any effects due to cross contamination.

Bulk hazardous waste containers are located away from the testing activities. Waste disposal uses lab pack procedures and those designated by the regulatory authorities. The Chemical Hygiene Plan and the Waste Management and Disposal SOPs (Westboro: SOP/14-01 and Mansfield SOP/G-006)) include the procedures for handling and disposing of chemicals used in the laboratory.

The working and storage environments are maintained in a safe and appropriate manner. A Chemical Hygiene Plan details the requirements for safety and chemical handling. Safety measures that protect property and personnel from injury or illness include: fume hoods, fire extinguishers, fire blankets, alarm systems, safety training, protective clothing, emergency showers, eyewashes, and spill control kits.

# 7.2 Work Areas

Good housekeeping is the responsibility of all personnel. Each person is to consible for assuring clean and uncluttered work areas. The job descriptions list spe ific housekeeping duties. Records, samples and waste materials are the common cause for clutted in the laboratory.

Management does not allow accumulation of boxed recc 1s, the aboratory operating area. Removal of administration and laboratory records to the rec rd storage area occurs to reduce clutter and ensure traceability. The individual filling the laberatory record box, labels the box with a number, the contents, date and laboratory area. A thorage area occurs to reduce of the box number, discard date and box contents. Authorized personnel review the box label for number, discard date and contents. Torget are stored on site and off-site for the record retention period identified in the NEL 20 at 1 EPA regulations, whichever is more stringent.

Sample management personnel removes to mplor to the sample storage area after all data is correct and complete. Sample cooler, are removed to a designated storage area for recycling. Samples are stored in the designated process storage areas until testing is complete. Sample removal from the process strage occurs after mailing of the final report. The sample management staff places the simples in the archive storage area for thirty days after report release. The archive sample storage storage is not controlled or monitored. Based on client specifications, samples are propely disposed or returned to the client.

Waste materials, e pired bagents, expired standards and materials are disposed of and not stored in the le praciny. Hazardous waste labeled accumulation containers in the laboratory collect designated vast streams for later bulk disposal. Laboratory personnel remove the less than five-g "lon accumulation containers when full from the laboratory and place the containers in the bulk hat rdou" waste area. Refer to the Waste Management and Disposal SOPS for Westboro: SOP/14-01 and Mansfield SOP/G006. Personnel identifying out of date reagents and standards remove the materials to the proper disposal area.

# 7.3 Security

Alpha Analytical provides a secure environment for our employees, guests, clients, samples and analytical data. Security procedures require that all exterior doors remain locked unless manned. Access to the laboratory is limited to employees and contractors. Visitors not under signed contract are required to sign the Visitors Log and must be accompanied by a laboratory employee at all times within the testing areas.

The defined high security area is the sample management area. Identification card locks on the internal doors control entry into the laboratory area.

All doors are locked after hours and require a key for entry. The security alarm continuously monitors for smoke and fire related heat. When the alarm is activated, the appropriate emergency response officers are notified. The local emergency offic s have the emergency contact list for the laboratory.

# 8 Equipment and Reference Materials

### 8.1 Maintenance

The laboratory has a proactive equipment maintenance program. The laboratory maintains service contracts for major equipment, which include routine preventative maintenance visits by the service provider. Technical personnel perform manufacturer's specified maintenance on a routine basis to ensure equipment operates at peak performance.

Procedures and schedules for preventive maintenance are available in the test method SOPs. A brief summary of some common preventive maintenance procedures `> provided in Appendix E. All instrument preventative and corrective maintenance is recorded in to > maintenance logbook assigned to the equipment. After maintenance or repair, the ins' ume. \* must successfully calibrate following the method SOP. Laboratory personnel must a monstrate quality control performance before sample analysis.

The laboratory maintains a stock of spare parts and contrum. Her for analytical equipment. Backup instrumentation for some analytical equipment is chailed to on site for use in case of major equipment failure. The person discovering or suspection and equipment maintenance problem or failure tags the equipment with 'out of service' tal. If notified and the appropriate equipment service provider is contacted.

All major laboratory equipment has individual and usceable maintenance logbooks in which to document manufacturer's recommender multiplication procedures, specific cleaning procedures, comments on calibration, replacement of small worn or damaged parts, and any work by outside contractors. The person performing routine or non-routine maintenance signs and dates the maintenance logbook. If an instrument is down for maintenance, a complete record of all steps taken to put it back into service is recorded including reference to the new calibration and quality control checks. Any equipment service providers working on the equipment are recorded in the logbook.

Record repetit. a or on-goin y equipment problems other than normal maintenance requirements on nonconformal. a action forms. The nonconformance action form notifies management and the Quality A surance of ficer of a problem affecting the performance and data quality.

The laborator, <u>resp</u> some equipment into a single laboratory equipment maintenance logbook. Examples include: autopipets, thermometer calibration. The identity of each item is by serial number or a laboratory-designated item number. The same data recorded for major equipment applies to this documentation.

The maintenance records shall include:

- Equipment name;
- Manufacturer's name, type identification, serial number or other unique identification;
- Date received, date put into service, condition when received;
- Current location;
- Details of past maintenance and future schedule;
- A history of any damage, malfunction, modification or repair;
- Dates and results of calibration or verification.

The maintenance logbook may include the reference to the location of the equipment operational and maintenance manuals. The logbook may include the reference to laboratory run logbook or data files for the calibration and quality checks of daily or frequent calibrations.

The Courier Supervisor ensures that maintenance and records for transportation vehicles are complete. The purchasing process is used for ordering garage maintenance, the garage work order is reviewed, and the vehicle checked for condition. The Controller receives all paperwork for completion of the maintenance process.

#### 8.1.1 Microbiology General Equipment Maintenance

Optics of the Quebec colony counter and microscope are cleaned prior to each use. The stage of the microscope is also cleaned and the microscope is kept covered when not in use.

Glassware is checked for residual alkaline or acid residue utilizing brathymol blue (BTB) on each day of media preparation.

# 8.2 Equipment Listing

A listing of the major equipment used for testing is available upon request. The equipment list details the unique identification number, equipment 'ocatic', serial number, model number, and purchase date. The unique identification number is all child to the piece of equipment.

The laboratory performs analyses using stable of the art equipment. In addition to the major equipment, the most common equipment, used the laboratory are: thermometers, balances, autopipets, water baths, hot plates, a poclave pH meters, conductivity meters and a variety of labware. The SOPs list the calibration and verification requirements for all laboratory equipment used in measurements.

# 8.3 Laboratory Wate

Laboratory water is pulified from central DI water systems and piped to all laboratory areas. In Westboru the QA Lapartment samples the laboratory grade water and submits the samples for analysis by the lab o document the water meets the drinking water certification criteria. The Laboratory Wuter Logbook lists the daily conductivity checks and acceptance criteria for the laboratory water. The laboratory documents the daily, monthly and annual water quality checks. Please refer to Table 8-1 for tested parameters, monitoring frequency and control limits for each parameter (SOP/08-11). Additional parameters may be tested for at the laboratory's discretion.

When additional treatment occurs in the test area, that test area records the water quality checks from the most frequently used tap. At a minimum the quality of the laboratory grade water is monitored daily by conductivity measurements. Records of the daily checks are found in the Laboratory Water Logbook. If out of specification results occur, a nonconformance action form is submitted.

# TABLE 8-1 (Westboro Water)

<u>Parameter</u>	Monitoring Frequency	Control Limits
Conductivity	Daily	<2 µmhos/cm @ 25°C
рН	Daily	5.5 - 7.5
Total Organic Carbon	Monthly	< 1.0 mg/L
Total Residual Chlorine	Monthly	< detection limit
Ammonia Nitrogen	Monthly	< 0.1 mg/L
Metals: Cd, Cr, Cu, Pb, Ni and Zn	Monthly (Required Annually)	< 0.05 mg/L
Total Metals	Monthly (Required Annually)	< 0.1 mչ 'l.
Heterotrophic Plate Count Westboro only	Monthly	< 500 JFU/r '.
Water Quality Test (Biosuitability) Westboro only	Annually	C.o - 3.0o

# 8.4 Reference Materials

Reference materials include: Class 1 weights, N. T thermometers and reference standards. Logbooks record the reference materials, see to calibration and verification. The Department Manager maintains any certificates received with the reference materials. Laboratory personnel record in the standards logbook the reference standards date received, unique identification number, expiration date and number of containers. Each laboratory area, records the unique identifier on the reference standard ertificate and the Department Manager maintains the certificate. The identifier all ws trace to utility from the certificate to the analytical data.

# 9 Measurement Traceability and Calibration

### 9.1 General Requirements

All measuring operations and testing equipment having an effect on the accuracy or validity of tests are calibrated and/or verified before put into service and on a continuing basis. The results are recorded in the instrument specific logbook. The laboratory has a program for the calibration and verification of its measuring and test equipment. The program includes all major equipment and minor equipment such as balances, thermometers and control standards. The Quality Systems Manual and method SOP describe the calibration records, frequency and personnel responsibilities.

# 9.2 Traceability of Calibration

The program of calibration and/or verification and validation is equipment is such that measurements are traceable to national standards, while evaluable. Calibration certificates indicate the traceability to national standards, provide the recults, and associated uncertainty of measurement and/or a statement of compliance with identified recults, and associated uncertainty of that provides traceability to a national standard calibration of reference standards. The laboratory maintains a permanent file of all such certifications.

## 9.3 Reference Standards and Mater als

Alpha Analytical has a program for libit, 'ion and verification of reference standards. The results and program are recorded in t e app opnate instrument logbook. Required in-service checks between calibrations and verific, 'ions are described in method SOPs and are recorded in the appropriate instrument log! ook.

Calibration standard an initial strictly to method required calibrations. A logbook of use is maintained an initial strictly to method required calibrations. Each calibration standard is identified as to use a limit of strictly to method received, date opened, and expiration date. Calibrations are verified by using a second source or lot number of the calibration standard. Calibration check procedures are state lin applicable test method SOPs.

Reference standards of measurement in the laboratory's possession (such as calibration weights or traceable thermometers) are used for calibration only and for no other purpose.

### 9.4 Calibration General Requirements

Each calibration record is dated and labeled with method, instrument, analysis date, analyst(s) and each analyte name, concentration and response. For electronic processing systems that compute the calibration curve, the equation for the curve and the correlation coefficient are recorded in the appropriate instrument logbook. This is also true for manually prepared curves.

Initial calibration requires a standard curve that brackets the expected sample concentration. Initial calibration generally uses three to five standards depending on the equipment and reference method specifications. Before the start of each analytical sequence, initial calibration is verified by using a continuing calibration standard. Calibration verification or continuing calibration uses a standard from a second source or lot number than that used for initial calibration. The acceptance criteria for the continuing calibration standard must meet acceptance criteria before analysis of any samples. When the acceptance criteria is not within limits, review maintenance protocols and perform any necessary maintenance before starting the initial calibration sequence.

# 9.5 Equipment Calibration

The SOP used for the analysis defines the instrument and equipment calibration required. The following defines the general practices for equipment calibration of selected equipment.

### 9.5.1 Gas Chromatography/Mass Spectrometry (GC/MS)

The GC/MS is hardware tuned before performing the initial and <u>section</u> calibrations. Results must meet the peak ratio specifications of the analytical retricter For volatiles analyses, bromofluorobenzene (BFB) is used, and for semivolatiles analyses, de afluorotriphenylphosphine (DFTPP) is used for instrument tuning.

The mass spectrometer response is calibrated by an alyze in a let of five or more initial calibration solutions, as appropriate, for each GC/MS method. Each olution is analyzed once, unless the method or the client requires multiple analyses. The the start of response factor for each analyte is calculated for internal standard calibration. The combration factor for external standard calibration is calculated using the expressions found in the laboratory method SOP. Calibration is acceptable when all acceptance criteria are within clans.

The initial calibration is verified throws hue analysis of a continuing calibration standard every 12 hours. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors for all analytes of interest are calculated and verified against the initial cultoration mean relative response factors. The percent difference (%D) for each analyte is calculated and nust be less than the acceptance criteria stated in the method.

An acceptable cost nuing calibration run must have measured percent differences for the analytes within a thot's specified ranges. If any criteria for an acceptable calibration are not met, either instrument resinted ance must be performed until the continuing calibration analysis meets all criteria continuing calibration is established before any samples are analyzed. No samples may be analy, ad these the acceptance criteria are met for the initial and continuing calibration.

Additional quality control samples are part of the GC/MS analysis. These include internal standards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spikes and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SOP.

### 9.5.2 Gas Chromatography (GC)

Internal standard calibration or external standard calibration is utilized for analysis by GC. The method-specified number of calibration standards is used. Each solution is analyzed once and the analyte relative response factors or calibration factors are calculated. The mean relative response factor for each analyte is then obtained by using the expression in the formula listed in the SOP. Integrated areas are utilized for these expressions.

For multiple response pesticides, PCBs or hydrocarbons the quantitation consists of the average of selected peaks or the integration of the area defined by a reference standard. The SOP details the integration criteria for each compound.

The initial calibration is verified through the analysis of a continuing calibration standard every 12 hours or 20 samples. The concentration of the continuing calibration standard is dependent on the requirements of the specific method. The relative response factors  $f_{1}$  all analytes of interest are calculated and verified against the initial calibration mean relative response factors. The percent difference (%D) for each analyte is calculated. The percent d<sub>1</sub> \* (%r) may be calculated when calibration factors are used for quantitation.

An acceptable continuing calibration must have measured percender of the analytes within method specified ranges. Should any content of the analytes within method specified ranges. Should any content of the continuing calibration not be met, either instrument maintenance is performing on the continuing calibration analysis meets all criteria, or a new calibration is established by fore any samples are analyzed. No samples may be analyzed unless the acceptance content are met for the initial and continuing calibration.

Other standard checks may be recorded, r a specified reference method. Instrument performance checks specified in the reference method must be performed and be within the acceptance limits stated in the reference method. Additional quality control samples are part of the GC analysis. These include in the reference dards, surrogates, method blanks, instrument blanks, laboratory control samples, matrix spike and matrix spike duplicates. The frequency and control criteria are defined in the laboratory SCP.

## 9.5.3 Cold Vapor Atom or hsorphone Spectrophotometry (CVAA)

An initial calibration, performed daily with freshly prepared working standards that bracket the expected concentration, range of the sample. A minimum of a three-point calibration curve is acquired, bich mus have a correlation coefficient of 0.995 or better. The initial calibration is verified even, 10 s mples. The continuing calibration is required to be within method-defined criteria, depending on the analytical method employed. Continuing calibration blanks are run at the same frequency. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within  $\pm$  10% of the true value.

### 9.5.4 Inductively Coupled Plasma Emission Spectrophotometry-Mass Spectrometry (ICP-MS)

Initial calibration and instrument tune is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of a minimum of three standards and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within method-defined criteria. The continuing calibration is required to be within method-defined criteria. Interference check standards are performed at the beginning of the sequence. Acceptance criteria are stated in the SOP.

#### 9.5.5 Inductively Coupled Plasma Emission Spectrophotometry (ICP)

Initial calibration is performed daily, not to exceed 24 hours, and continuing calibrations are performed every 10 samples. Initial calibration consists of one standard and a Blank that bracket the expected concentration range of the samples. Analysis of samples cannot begin until an initial calibration verification has been performed and is found to be within 5% of the true value. The continuing calibration is required to be within 10% of the true value. Interference check standards are performed at the beginning and end of the sequence. Acceptance criteria are stated in the SOP.

#### 9.5.6 Thermometers

Laboratory thermometers are checked annually for accuracy against certified, NIST traceable thermometers. Correction factors derived from the annual calibrations applied to temperature readings where applicable. The analyst records the corrected temperature for all observations.

NIST traceable thermometers are calibrated professionally and re-cei field  $\epsilon$ -ary year. Records of thermometer calibrations are retained by the QA Departmer ..., "u. ...ometers are tagged with the ID number, correction factor to be applied and the expirition of the calibration check.

**NOTE:** Electronic-based thermometers are calibrated on  $\gamma$  quarterly basis by an outside instrument service company. Thermometers are tagged with calibration information by the vendor, including the ID number, correction factor to  $\gamma \rightarrow \alpha \beta$  pieled and the expiration of the calibration check. Certificates are kept on file in the CA Department.

Thermometers are not used past the calle ation, expiration date or if the thermometer is not reading properly. Replacement thermometers are calibrated and the maintenance logbook is updated when a change in the thermometer is required due to breakage, damage or expired calibration.

### 9.5.7 Balances

Calibration checks are per primed for each day of use, for each balance. The calibration consists of a minimum of two weig. ts, w ich bracket the weight to be measured. Additional calibration check procedures ar pe formed on balances utilized in Microbiology laboratory. This additional procedure condists date, ction test, which is performed to ensure that 100mg is detectable at a weight of 150 g. ms.

The balance logbook lists the acceptance criteria and performance criteria for the various balances use in the laboratory. Calibration weight measurements must meet the acceptance criteria listed on the record form.

Each balance is serviced and calibrated by a professional semi-annually. Balances are labeled with the balance number, date of service and the expiration date for the annual service check. The balance number used for any measurements requiring traceability is recorded with measurement data. Balances are not used past the expiration date or when the weight check is not within acceptable criteria. The accuracy of the calibration weights used by Alpha Analytical is verified annually by an accredited calibration service.

#### 9.5.8 Automatic Pipettes

Delivery volumes for the automatic pipettes are checked and recorded gravimetrically before use and on a monthly basis. The verification is performed at the volume of use or bracketing the volume range of use. The check must be within the criteria stated in the laboratory logbook.

Autopipette calibration is also performed once per year. Each pipette is checked throughout the volume range of use by measuring seven replicate volumes and weighing. Acceptance criteria for

continued use is 5% RSD and between 95.0-105 % recovery. Pipettes failing acceptance criteria are tagged and removed from service until repaired and the criteria are met, or discarded and replaced. Automatic pipettes are labeled with a unique ID number, volumes verified and expiration date.

#### 9.5.9 Ion Chromatography

The ion chromatograph calibration is by analyzing a set of five or more initial calibration solutions, with concentrations of analytes appropriate to the analytical methods. The concentrations must bracket the expected concentration range of the samples analyzed. Procedures for verifying the calibration curve are method specific. The initial calibration is performed at the start of each day. The calibration curve is verified at least after every 20 samples.

#### 9.5.10 pH Meters

pH meters are calibrated prior to use for each day of use. The meter is calibrated following the procedure for pH analysis. The records of the calibration are recorded in an instrument logbook or in the raw data for the analysis being performed. At least two puffor solutions that bracket the measurement range for the analysis are used for calibration. A record source check standard is used at the end of a run to verify meter stability. Buffer solutions used for calibration are NIST certified. Standard buffer solutions are not retained or recuse. The lot number of the buffer solutions is recorded in the data record to ensure transactive cite measurement to NIST.

#### 9.5.11 Conductivity Meters

Three calibration standards of potassium chic. de (r CL) solutions are analyzed annually on each instrument range. The calibration starta, 's a, used to verify instrument performance. The acceptance criteria are defined in the st SCC If unacceptable performance is found, the cell is cleaned and rechecked. The cell is not used until satisfactory performance is achieved.

A single KCL standard solution is use I to calibrate each range of the instrument. A second standard is used to check the calib. Son each day the meter is used. The check standard is near the measurement range for the simples to be analyzed. The acceptance criteria is  $\pm 20\%$  of the true value. The meter is labeled with expiration date for the annual calibration. A check standard that is NIST traceal to allow traceability. The check standard is performed at the end of the analysis run that at the every 20 samples.

### 9.5.12 Autoclav

The date, contents, sterilization time and temperature, total cycle time and analyst's initials are recorded each time the autoclave is used. Autoclave cycles must be completed within 45 minutes when a 15 minute sterilization time is used. Autoclave timing mechanisms are checked quarterly with a stopwatch to verify timing controls. A maximum temperature thermometer is used with each cycle to ensure the sterilization temperature is reached.

Spore strips or ampoules are used weekly to confirm sterilization. BTSure ampoules are utilized as follows: An indicator ampoule is placed in most challenging area of sterilizer. Load is processed according to standard operating instructions. Remove from sterilizer and allow to cool for a minimum of 10 minutes. (Chemical indicator on label changes from green to black when processed.) Place the autoclaved indicator and un-autoclaved control indicator in an upright position in the plastic crusher provided. Gently squeeze crusher to break glass ampoules. Incubate both indicators at 55-60°C for 48 hours. Examine appearance for color change. Yellow color indicates bacterial growth. No color change indicates adequate sterilization.

Calibration is conducted and certified annually by an outside service provider and recorded. Certificates are kept on file. Routine maintenance includes cleaning the autoclave seal to ensure

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freedom of caramelized media and cleaning drain screens to remove any debris buildup. For the efficient operation of the unit, overcrowding is avoided.



# 10 Test Methods and Standard Operating Procedures

## 10.1 Methods Documentation

Analysis consists of setting up proper instrument operating conditions, executing acceptable calibrations, monitoring instrument performance tests, analyzing prepared samples, and collecting data from the analyses. The test method SOP describes the instrumental analysis procedures, quality control frequencies and acceptance criteria. EPA accepted methods, national recognized methods or client-specified methods are the basis for performance criteria, instrument conditions and the steps of the procedure. The method performance requirements of the published methods are followed unless otherwise specified by the client.

The reference methods define the instrument operating conditions. In n, ny of the reference methods, a range or general guidance on the operating condition, is draned. Documented modifications to the operating conditions clarify the reference method modifications are dener the performance criteria from the reference method must be met. Modifications to the operating conditions are stated in the SOP. Changes in the operating conditions made at tr. tin, of the analysis are documented in the appropriate laboratory or sequence log. A revision to be SOP takes place, when a day to day change in the operating condition improves performance to all matrices.

The laboratory SOPs include the operation of no asurement equipment. The SOPs contain the - following information, as applicable:

- The equipment used in the proc. dure, including equipment type
- Equipment calibration and , rocess for obtaining the measurement from the calibration
- The step by step ins uction, to perform the measurement
- Acceptance cri eria for use calibrations
- Corrective act. n for failed acceptance criteria, including assessment of previous calle attom results
- The bas, used for the calibration standards such as traceability to NIST or EPA e der. Instration of comparability
- Frequer y at which the equipment will be calibrated, adjusted and checked
- T. Ste ords maintained to document the calibration and use of measurement equipment
- The calibration status for the equipment
- The environmental conditions necessary before measurement equipment may be calibrated or used for measurement
- Allowed adjustments to measurement equipment, including software, which will not invalidate the laboratory analysis
- Maintenance of the equipment and record keeping to track performance before and after maintenance is completed
- Define the standards, reagents and sample handling, interferences, preservation, and storage in order to assure measurement performance

# 10.2 Standard Operating Procedures (SOPs)

Alpha Analytical maintains SOPs that accurately reflect all phases of current laboratory activities such as assessing data integrity, nonconformance actions, handling customer complaints, sample receipt and storage, purchasing of all materials, and all test methods. These documents include

equipment manuals provided by the manufacturer, internally written documents, and published methods with documented changes or modifications.

Copies of all SOPs are accessible to all personnel in either electronic or written form. The SOPs are organized in a standard format with the signatures of the approving authorities. Each SOP clearly indicates the effective date of the document and the issue number.

# 10.3 Laboratory Method Manual (s)

All SOPs are posted as secure documents on the Alpha Intranet. Directories are available for each laboratory and administrative area. Each SOP includes or references where applicable:

- 1) identification of the test method and where applicable;
- 2) applicable matrix or matrices;
- 3) method detection limit;
- 4) scope and application;
- 5) summary of method;
- 6) definitions;
- 7) interferences;
- 8) safety;
- 9) equipment and supplies
- 10) reagents and standards
- 11) sample collection, pre. rvatio. shipment and storage;
- 12) quality control;
- 13) calibration and s. ndar ization;
- 14) procedure
- 15) calcı atıc יs;
- 16) heth d performance;
- 17) poil tion prevention;
- . `) dat assessment and acceptance criteria for quality control

measurements;

- 19) corrective actions for out-of-control data;
- 20) contingencies for handling out-of-control or unacceptable data;
- 21) waste management;
- 22) references; and
- 23) any tables, diagrams, flowcharts and validation data.

In cases where modifications to the published method have been made by the laboratory or where the referenced method is ambiguous or provides insufficient detail, these changes or clarifications are clearly described in the SOP.

# 10.4 Test Methods

The laboratory uses appropriate methods and procedures for all tests and related activities within its responsibility (including sampling, handling, transport and storage, preparation of items, estimation of uncertainty of measurement and analysis of test data). The method and procedures are consistent with the accuracy required, and with any standard specification relevant to the calibrations or tests concerned. When the use of mandated methods for a sample matrix is required, only those methods are used. Where methods are employed that are not required, the methods are fully documented and validated and are available to the client and other recipients of the relevant reports.

The client requests the reference method for sample analysis usually based on the regulatory program. The client services staff may assist the client with method selection when the client specifies the regulatory program, but is unsure of the correct method equired. The Laboratory Director or Quality Assurance Officer recommends methods for non-regulatory programs. In all cases, recommendation of methods is based on client-defined method erformance criteria. Client services may recommend a procedure that meets the client method performance criteria.

# 10.5 Method Validation/Initial Demonstration of Moth. d ) for nance

Before acceptance and use of any method, satisfact ry i itial demonstration of method performance is required. In all cases, appropriate ..... a. completed and retained by the laboratory and made available upon request. All associated supporting data necessary to reproduce the analytical results is retained. If itial demonstration of method performance is completed each time there is a significant cleage in instrument type, personnel or method.

### 10.6 Sample Aliquots

The aliquot sampling process fror sul mitted sample is part of a test method. The laboratory uses documented and appropriate pricedures and techniques to obtain representative sub-samples. Sample aliquots removed for analysis are homogenized and representative portions removed from the sample container. Fersonnel record observations made during aliquot sampling in the test method logbook.

# 10.7 Data Verifica. on

Calculatic s and da a transfers are subject to appropriate checks. A second person recalculates all manual c. culatic is. An independent qualified analyst also reviews the data. A Client Services representative . ...ews data for project and method performance requirements where applicable. A QA representative reviews data for project and method performance requirements when requested by a Client. Final report review is performed by an authorized company signatory.

For drinking water suppliers, every effort is made to notify the Client within 24-hours of obtaining valid data of any results that exceed any established maximum contaminant level or reportable concentration. Analyst or Department Supervisor notifies the Client Services Department of the sample number(s), Client name, analysis and sample results (preliminary or confirmed). The Client Services Department notifies the client.

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The laboratory Report Generation and Approval SOP describes the practices to ensure that the reported data is free of transcription errors and calculation errors. Manually entered data into the LIMS is dual entered and checked by the LIMS to minimize transcription errors. The laboratory test method SOP describes the quality control measures used to assure method performance before reporting data.

# 10.8 Labeling of Standards and Reagents

The purchase, receipt and storage of consumable materials used for the technical operations of the laboratory include the following:

- a) The laboratory retains records of manufacturer's statement of purity, of the origin, purity and traceability of all chemical and physical standards.
- b) Original reagent containers are labeled with the date opened and the expiration date.
- c) Detailed records are maintained on reagent and standards prepartion. These records indicate traceability to purchased stocks or neat computing and include the date of preparation and preparer's initials.
- d) Where calibrations do not include the gener. 'ion f a ali', ation curve, records show the calibration date and type of calibration star. 'aro 'sed.
- e) All prepared reagents and standards a g un rue. identified and the contents are clearly identified with preparation date concent ation and preparer's initials.

### 10.9 Computers and Electronic Data Revised . `equirements

Computers or automated equipment are use for the capture, processing, manipulation, recording, reporting, storage or retricial of ast data. The laboratory ensures that computer software is documented and ademate. The goals of the software development methodology, existing system validations and the charge control system are to ensure that:

- the software systems perform the required functions accurately,
- the users understand now to use the system, and
- auditors .an . sure themselves of the validity of the analytical data.

The computer systems sed at Alpha Analytical are purchased. A coordinated effort is made with the support to asture the computer operations meet the laboratory requirements for data integrity. Alp a Ana' tical has a formal validation program of its computer systems. The validation program is a comprehensive program to ensure data transmitted, reported or manipulated by electronic means is correct and free of errors. The validation and verification approach is separated into three areas.

- New software is developed and validated using test data. Records of validation include the test data report, date and initials. Where formulas are part of the program, documentation includes manual verification of the final calculated values. New software includes the development of macros for spreadsheets and other tools using commercial software packages.
- 2. Reasons for changes to software are identified through flaws in existing documentation or the need to improve system processes and are documented on the Nonconformance Report. Final implementation of the change is documented on the nonconformance action form. The tracking and timelines of making the change is readily available. This process also provides the complete documentation of all software and electronic data reporting problems.

3. Verification of system integrity is through routine maintenance, protection from unauthorized access and electronic verification programs. Routine maintenance including system backups are performed on a scheduled basis. The backup process and password and access protections are defined in the Computer System Backup Control SOP/11-01 and Computer Security SOP/11-02. Electronic verification may be used to assure the commercially purchased software is performing at its original specifications. This includes virus checking of all network operation at least once per week. Documentation of all verification and maintenance operations is retained.

# 11 Sample Handling, Sample Acceptance Policy and Sample Receipt

The Sample Login and Custody procedures define the process for sample management from sample receipt through analysis and to disposal. These procedures detail the process for sample receipt, records and storage pending analysis.

Clients or Alpha's Courier service deliver samples to the laboratory during normal business hours. Sample receiving occurs in the sample management area.

Client service personnel place bottle orders. The orders are filled following the bottle order instruction form. Blanks are prepared as needed with minimal storage. All glass containers are wrapped in bubble wrap or mesh sleeves to prevent breakage. The ontainers are placed in plastic coolers or shipping packages and Chain-of Custody forms, some and labels enclosed. The bottle order is shipped by third party, picked up by the client or clie + representative or delivered by Alpha courier to the client.

## 11.1 Sampling Supplies

#### 11.1.1 Sample Containers

Sample containers provided by Alpha Analytical i alu 'e lab Is, preservatives and a blank chain of custody form. Preservatives and containers are at consolled and verified as appropriate for the indicated type of analysis.

Each lot of containers used for the collectic of samples for microbiological analysis is checked for sterility prior to distribution. Sterility checke are performed by Microbiology staff and results recorded in Microbiology Sample Society Sterility Log.

#### 11.1.2 Chain of Custody

Chain of custody forms multi accompany all samples received by Alpha personnel. The chain of custody form indicates the sample origin and arrival at the laboratory and identifies the analyses requested.

#### 11.1.3 Reager Water

Alpha Analytic... supplies laboratory pure water for field QC blanks. Water used for volatile organics must be free of volatile compounds below the method detection limit. The quality of the laboratory water is monitored for conductivity once per day. Additional water quality criteria may be monitored based on client specific requests. The water quality in the laboratory is monitored for chemical parameters as required by the EPA certification manual for drinking water (Water Quality Monitoring SOP/08-11).

### 11.2 Sample Tracking

Alpha Analytical routinely uses an internal chain-of-custody in LIMs for sample tracking control purposes. When requested or required by regulation a legal custody program is used in addition to the routine laboratory practices. Legal custody practices must be arranged at the time of contractual commitment.

For legal custody the process must include complete and continuous records of the physical possession, storage, and disposal of sample containers, collected samples, sample aliquots, and sample extracts or digestates. For legal custody a sample is in someone's custody if:

- 1. It is in one's actual physical possession;
- 2. It is in one's view, after being in one's physical possession;
- 3. It is in one's physical possession and then locked up so that no one can tamper with it;
- 4. It is kept in a secured area, restricted to authorized personnel only.

The routine sample handling and tracking process includes unique identification of all sample containers, initials of the person removing the sample from the sample management area and documentation of the date of sample removal for disposal.

Samples are assigned a unique identification number from the LIMS program. Each sample container label includes a unique identifier for the container. The person handling the sample is recorded along with the unique identifier in the container tracking records in LIMS.

ALPHA ANALYTICAL utilizes a custom designed Laboratory Inform tion N nagement System (LIMS) to uniquely identify and track samples and analytical date the distribution of the facility. The LIMS log-in, is initiated by the Sample Custodian when the following information is entered into the computer:

- Quote number (unique to the project)
- Project name or description
- Analyses requested (per matrices receive ')
- Sample number (unique to this sample)
- Sample descriptions (client ID, incluing rumber of received containers)
- Date received
- Date(s) and time(s) collected
- Date analytical results are due
- Client's name and address
- Notation of special handli g inst. ctions
- Additional comments or illistruction for the laboratory
- Purchase order nu iber(s), in applicable

Alpha Job Numbers (r) ceal or assigning numbers)

Alpha Job Numbe, are unique #'s automatically designated by our LIMS completer system for every individual client project.

There are `nr .s to this number:

- All numbers start with the letter "L"
- The next two numbers are the last two numbers of the current year.
- The last five numbers are pulled sequentially by the LIMS as each Login personnel requests a new number for a job.

For example.... L0904165 ---- Year 2009 and 4,165<sup>th</sup> job to be logged in this year.

The Alpha Job Number then may contain as many extensions as there are individual samples in a job. L0904165-01 is the first sample, L0904165-02 is the second and so on. Each sample may contain as many as 26 containers as the containers are designated with the letters of the Alphabet, and each container receives it's own barcoded label. For example, L0904165-09A is the first container of the 9<sup>th</sup> sample listed on a client's Chain of Custody.

Each container is labeled with a unique identifier, a label with a unique identifier number is placed on each sample container. Once labeled, the sample containers are placed in the appropriate storage area.

# 11.3 Sample Acceptance Policy

The sample management personnel check for proper sample labeling, preservation and handling at the time of arrival at the laboratory. The client and client services manager specifies the proper sample preservation, containers, cooling and other criteria on the project review form and in the LIMS. Sample management staff record all observations and immediate notify client services of any discrepancies or questions arising during sample receipt.

It is possible for samples or sample containers to be lost, damaged, or determined to be unsuitable, for whatever reason, after initial receipt at Alpha Analytical. Should this happen, the event is recorded on the nonconformance action form by the observer free problem is brought to the attention of a client services manager who reports it to the client. Plans or disposition of the affected samples or container are agreed upon with the client, corried out, and recorded in the project records.

# 11.4 Sample Receipt Protocols

The sample management staff receives all sample . A u. ique job number is assigned to each shipment of samples received from a client. The induce excords for the incoming job, including the internal Chain-of -Custody, are initiated with. Sam, belivery Group (SDG) form. The client and the sample management personnel signer hes, mple custody form at the time of receipt at the laboratory. Samples received via overnight currier are signed on the bill of lading. The bill of lading, SDG form and the sample culitor, form are completed for external courier delivered samples.

The sample management st ff ex mines the shipping containers, their contents, and accompanying client documentation. I formation about the sample identification, the location, date and time of collecting, collector's name, preservation type, sample type, presence and condition of custody seals, he state of preservation of the samples and other required information is noted on the SEG rm. Any discrepancies in documentation or problems with sample condition such as a proprix the sample containers, thermal preservation variation, holding times and adequate supple volumes are noted and brought to the attention of the client via the nonconformance a tion form. The Client Services Manager provides clarification or further instruction, the sample management staff on the processing of the samples that are incomplete or missing require information.

The sample management staff logs the samples in the LIMs and a durable label for each container is printed. The custodian attaches each label to the appropriate sample container. The following information is recorded for tracking internal custody: laboratory sample ID, client sample ID, sample matrix and storage location. Sample receipt and log-in specifically requires: date and time of laboratory receipt of sample(s); sample collection date; unique laboratory ID code; field ID code supplied by sample submitter; requested analyses; signature or initials of data logger; comments from inspection for sample acceptance or rejection and in some cases, sample bottle codes.

# 11.5 Storage Conditions

Alpha Analytical stores samples under proper environmental conditions to ensure their integrity and security. Samples are stored at temperatures that meet specifications of the methodology, regulatory agencies and client directives. Refrigerators are monitored and controlled to be within  $4 \pm 2^{\circ}$ C. Chemical, temperature, holding times and container storage requirements are listed in the LIMS project database.

Client Quality Assurance Project Plans may list preservation requirements differing from the laboratory. The sample management staff reviews project information for projects specific handling. Addition of chemical preservative to sample containers normally is done in the field at the time of sampling. Chemical preservation and temperature preservation checks at the time of receipt are recorded except for volatile organic compounds, bacteria, sulfite, and dissolved oxygen preservation. Any differences from laboratory or client specific requirements are recorded on nonconformance action forms and contact made with the client by the Client Services Manager or designee.

Sample storage facilities are located within the sample management area, which is secured by locked doors. Internal chain-of-custody procedures and documenta, on pertaining to sample possession, removal from storage, and transfer are outlined in the sense le custody procedure. Samples are returned to the sample management area after the sample posterior is removed for analysis. Extracts and digestates are tracked and follow the same is ternal custody operation. Extracts and digestates are removed to the waste disposal area after unalysis for proper disposal.

Sample storage precautions are used to ensure that  $c_1$  as  $c_2$  in the simulation does not occur during sample storage. Refrigerator storage blanks are monitored for totalile compounds as necessary. The storage blank information allows the assessment of  $\mu$  tential cross contamination in the sample storage refrigerator.

Temperatures of cold storage areas are minitorial and recorded daily. Corrective action is done as necessary when temperatures are not will in this control criteria. Temperatures are measured with NIST traceable thermometers. All the periture records indicate the thermometer used for obtaining the measurement. In the Wespord facility, the Data logger is linked to thermocouples in several refrigerators and freezers in the Sample Storage area. The Data logger is calibrated and certified by an outside vendor on a quitter.

# 11.6 Sample Disposal

Samples are held for the try for after the report is released to the client. Upon written client request samples are held for up to six months in an uncontrolled area. Requests for controlled sample storage. Just a arranged at the time of contractual commitment

An authol. ed wast carrier is contracted to pick up waste as needed and dispose of it, in accordance with all regulatory requirements. Post-analysis disposition of samples is dependent upon project specific requests. Remaining sample material may be returned to the client, safely discarded, or archived for a specific time prior to disposal. The waste disposal SOP defines the specific requirements for sample disposal and other waste disposal operations.

The sample management staff are responsible for the archival and disposal of raw samples, extracts and digestates. Raw and prepared samples may not be archived or disposed until all of the designated analyses are complete and resultant analytical data is sent to clients. Samples in storage are retained a minimum of 30 days after reporting the results to the client. Any samples requiring more than 30 days are archived.

When a client has requested the return of samples, the sample management staff prepares and ships the samples according to the same custody procedures in which the samples were received and following any client specified requirements. Protection of the samples during delivery is ensured by the implementation of special packaging procedures. Packages are delivered by a commercial carrier whose procedures for protecting the samples are not within the control of this laboratory. Clients are informed that a commercial carrier will deliver their samples if required.

### 12 Records

Alpha Analytical has a record system that produces unequivocal, accurate records, which document all laboratory activities. The laboratory retains records of all original observations, calculations and derived data, calibration records and a copy of the test for ten years minimum. The system retains records longer than the minimum upon the request of authorized clients, agencies or another regulator.

# 12.1 Record Keeping System and Design

The record keeping system allows reconstruction of all laboratory processes that produced the analytical data of the sample.

- a) The records include the names of personnel involved in sampling, preparation, calibration or testing.
- b) All information relating to laboratory facilities <u>viii</u> ant, analytical methods, and activities such as sample receipt, preparation, or <u>lata</u> articles and accumented.
- c) The record keeping system guarantees retrieved of a 'working files and archived records for inspection and verification purposes.
- d) All documentation entries are signed or initiale the responsible staff.
- e) All generated data requiring opera\*. logs on appropriate logsheets or logbooks are recorded directly, promptly and legibly, permanent ink
- f) Entries in records are not obiligrated by any method. All corrections to errors are made by one line marked through the cror. The person making the correction signs and dates the correction.
- g) Data entry is minimized we electronic data transfer and ensuring the number of manual data transcriptions is reduced.

### 12.2 Records Management and Storage

- 1. All reards including calibration and test equipment, certificates and reports are safe / stored, held secure and in confidence to the client.
- 2. I a Loratory maintains all hardware and software necessary for reconstruction of data.
- 3. Records that are stored or generated by computers have hard copy or writeprotected backup copies.
- 4. Alpha Analytical has established a record management system, for control of all hard copy laboratory notebooks.
- Access to archived information is carefully controlled and is limited to authorized personnel. These records are protected against fire, theft, loss, environmental deterioration, vermin, and in the case of electronic records, electronic or magnetic sources.
- 6. In the event that Alpha Analytical transfers ownership or goes out of business, there is a plan to ensure that the records are maintained or transferred according to the client's instructions. A shell corporation will be developed to maintain continuity of our record keeping systems as requested and/or required by both state and federal laws.

Alpha Analytical retains all original hard copy or electronic raw data for calibrations, samples, and quality control measures for ten years, including:

- 1. Analysts work sheets and data output records,
- 2. Reference to the specific method,
- 3. Calculation steps including definition of symbols to reduce observations to a reportable value,
- 4. Copies of all final reports
- 5. Archived SOPs,
- 6. Correspondence relating to laboratory activities for a specific project,
- 7. All nonconformance action reports, audits and audit responders,
- 8. Proficiency test results and raw data,
- 9. Data review and cross checking.

The basic information to tie together analysis and peripherals uch as strip charts, printouts, computer files, analytical notebooks and run logs for Alpheral Ivite duraludes:

- 1. Unique ID code for each Laboratory sam' .e. Qc sample;
- 2. Date of analysis;
- 3. Instrument identification and operating conditions;
- 4. SOP reference and version;
- 5. Any and all calculations;
- 6. Analyst or operator's inuc's/s. nature.

In addition, Alpha Analytical ... in. ins jecords of:

- 1. Personnel qual "catic is, experience and training
- 2. Initial a I contuing demonstration of proficiency for each analyst
- 3. A log of names, initials and signatures for all individuals who are responsible for sign. g or initialing any laboratory records.

### 12.3 Laboratory Sample Tracking

A record of all procedures to which a sample is subjected while in the possession of the laboratory is maintained. These include but are not limited to all records pertaining to:

- a) Sample preservation including appropriate sample container and compliance with holding time requirement; If the time of the sample collection is not provided, the laboratory must assume the most conservative time of day (i.e., earliest).
- b) Sample identification, receipt, acceptance or rejection and log-in;
- c) Sample storage and tracking including shipping receipts, transmittal forms, and internal routing and assignment records; this includes interlaboratory transfers of samples, extracts and digestates.
- d) Sample preparation including cleanup and separation protocols, I code volumes, weights, instrument printouts, meter readings, calculations, reagents;
- e) Sample analysis;
- f) Standard and reagent origin, receipt, preparation, and cie;
- g) Equipment receipt, use, specification, operating onditions and preventative maintenance;
- h) Calibration criteria, frequency and acceptone criteria;
- i) Data and statistical calculations, ... iew, confirmation, interpretation, assessment and reporting conventions;
- i) Method performance criteria inclusing spected quality control requirements;
- k) Quality control protocol and coser ament;
- I) Electronic data securit, sof ware documentation and verification, software and hardware audits, backups, and , coros of any changes to automated data entries;
- m) All automated ram, 's handling systems;
- n) Records storage and retention; and
- o) Disposal of hazardous samples including the date of sample or sub-sample disposal and the name of the responsible person.
- p) The COC records account for all time periods associated with the samples.
- q) The COC records include signatures of all individuals who had access to individual samples. Signatures (written or electronic) of all personnel who physically handle the samples. Time of day and calendar date of each transfer or handling procedure
- r) Common carrier documents.

### 13 Laboratory Report Format and Contents

The Process Planning and Control Procedure details the recording and reporting of data as required by the client and in accordance with relevant environmental regulations.

Clients specify the report delivery and deliverables required for the work submitted. Report delivery includes standard turnaround and rush turnaround. Clients specify the delivery address or multiple addresses and method of delivery such as U.S. Mail, facsimile or electronic at the start of the project. Alpha Analytical provides data deliverables in hardcopy or electronic format. At the start of any project, the electronic deliverable formats required must be received before sample arrival.

Reporting packages are available for routine regulatory reporting , guirements. Regulatory reporting packages include only the information requested by the regulatory agency. In addition to regulatory report packages, Alpha Analytical prepares a standard report to mat. The standard report format includes:

- 1. Title: "Certification of Analysis"
- 2. Name and address of the laboratory
- 3. Laboratory Job Number, page number a ש ני אור דו mber of pages included in the report.
- 4. Name and address of the clien
- 5. Alpha sample number, Client ider, 'fication, Sample location
- Samples identified that do .ot n. et the sample acceptance requirements for project.
- 7. Date of sample recept, sa. ple collection, analysis date and time, report date and analyst
- 8. Identification c data ported by subcontractors
- 9. Test nam , d L ^ , eference method number
- 10. De. ery vethod and sampling procedures when collected by lab personnel
- 11 Deviations of modifications that affect data quality
- 12. \*atem/ it that results relate only to the sample tested
- **13.** Statement that report must be copied in full unless the laboratory provides written permission for partial copies
- 14. Glossary, References and limits of liability
- 15. Units of measure and reporting detection limit
- Quality control data for: % Recovery surrogates, % Recovery of LCS, % RPD of LCSD, Blank analysis, % Recovery Matrix Spike, %RPD of Laboratory Duplicates, as applicable
- 17. Signature, title and date of report

Results transmitted by facsimile or other electronic means include a statement of confidentiality and return of the materials at the laboratory's expense.

The laboratory notifies the client in writing of any circumstance that causes doubt on the validity of the results. The amended or modified report lists the change, reason for the change, affected page numbers, date of the amendment and authorized signature.

### 14 Outside Support Services and Supplies

When Alpha Analytical purchases outside services and supplies in support of tests, the laboratory uses only those outside services and supplies that are of adequate quality to maintain confidence in the tests.

The Purchasing SOP/13-01 describes approval and monitoring of all suppliers and subcontractors used by the laboratory. Where no independent assurance of the quality of outside support services or supplies is available, the laboratory ensures that purchased equipment, materials, and services comply with specifications by evaluating method performance before routine use.

The laboratory checks shipments upon receipt as complying with purple = specifications. The use of purchased equipment and consumables is only after the evaluation  $a_i + compliance$  to the specifications is complete. The Purchasing SOP/13-01 describes the definition of purchased product.

The Purchasing SOP describes the process for raish a review and placement of purchase orders. It is company policy to purchase from third party artin, d suppliers and subcontractors wherever possible. Purchases must be from supplier, ap, row d by the Laboratory. Laboratory or sampling subcontractors specified by the client are noted is "Trial" on the purchase order. This identifies the subcontractor as a non-approved support fractor.

The laboratory maintains list of approved ventions (, orm 13-01) and subcontractors from whom it obtains support services or supplies required for in ts.

### 14.1 Subcontracting Analytical Jan The

Clients are advised, verbally and for in writing, if any analyses will be subcontracted to another laboratory. Any testing contracted under NELAC that requires subcontracting, will be subcontracted to another NELAC accredined laboratory for the tests to be performed and any testing covered under the DOD QSN that requires subcontracting, will be subcontracted to another accredited DOD laborated and musicine project-specific approved from the DOD client before analysis begins. These requires for DOD projects pertain to both Westboro and Mansfield facilities. The laboratory app. Wes testing and sampling subcontractors by review of current state, national or other examples are careful and samplings. This document must indicate current approval for the subcontract of work.

The Sample Receipt and Login Procedure describes the process for sample handling when subcontracting samples. The quotation form lists the subcontractor in order to notify the client of any subcontracted work. Client notification of subcontracted work is in writing before releasing samples to the subcontractor.

The review of subcontractor documents for completeness and meeting the specifications defined for the project follows the laboratory process for reporting and verification of process data. The person responsible for receiving the order reviews the information supplied by the subcontractor instead of the Department Supervisor

### 15 Client Relations

### 15.1 Client Service

The majority of the client services occur from personnel in the administration, sample receiving and sampling areas. Client service involves inquiries into services offered, technical consulting, placing orders, and receiving orders, providing updates on the status of orders and completing orders. Personnel interacting with clients must document and review client specific project requirements. Call Tracker is used to document communications with clients (SOP/10-02). Personnel must document client interactions following the appropriate laboratory procedures. Each person must communicate deviations, modifications and client requests following the laboratory defined procedures.

### 15.2 Project Management

During staff meetings the laboratory management reviews requests for new work. The Operations Director and/or Laboratory Director addresses al. cupacity and capability issues. Where conflicts in workload arise, client notification is mm. Hat in Project Communication Form (PCF) contains the documentation of all project in tration. Cooperation between laboratory and client services staff allows direct count mice ion and scheduling. Management arranges complex scheduling and coordination betw en de, artmental areas.

### 15.3 Complaint Processing

The laboratory staff documents all clier is cloth, parties' complaints or concerns regarding the data quality or laboratory operations. I a Non-onformance Report records complaints, correcting the concern, and resolving the concern with the client or other party. The process uses the same form and process as the nonconformince action process. Where repetitive corrective actions indicate a problem, an audit of the area, Customer Inquiriy and Complaint SOP/10-01 is immediate to ensure the corrective action has effectively solved the concern.

### 16 Appendix A – Definitions/References

The following definitions are from Appendix A of the 2003 NELAC Standard. The laboratory adopts these definitions for all work performed in the laboratory.

- Acceptance Criteria: specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)
- Accreditation: the process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory. In the context of the National Environmental Laboratory Accreditation Program (NELAP), this process is a voluntary one. (NELAC)
- Accrediting Authority: the Territorial, State or Federal agency havin, responsibility and accountability for environmental laboratory accreditation divide grants accreditation. (NELAC)[1.4.2.3]
- Accrediting Authority Review Board (AARB): five oting memory from Federal and State Accrediting Authorities and one-non-voting memory is m USEPA, appointed by the NELAP Director, in consultation with the NELAC Board of Directors, for the purposes stated in 1.4.7.e (NELAC)[1.4.7]
- Accuracy: the degree of agreement be eer, an observed value and an accepted reference value. Accuracy includes a combination of rundom error (precision) and systematic error (bias) components which are due to sampling and analytical operations; a data quality indicator. (QAMS).

Aliquot: A discrete, measu. vd, representative portion of a sample taken for analysis. (DoD; EPA Q/ J gloss. , )

- Assessor Boc the organization that actually executes the accreditation process, i.e., receives and reviews accreditation applications, reviews QA documents, reviews proficiency 'estil g results, performs on-site assessments, etc., whether EPA, the State, or ontracte private party. (NELAC)
- Analyst: the designated individual who performs the "hands-on" analytical methods and associated techniques and who is the one responsible for applying required laboratory practices and other pertinent quality controls to meet the required level of quality. (NELAC)
- Analyte: The specific chemicals or components for which a sample is analyzed; it may be a group of chemicals that belong to the same chemical family, and which are analyzed together. (EPA Risk Assessment Guide for Superfund; OSHA Glossary)
- Applicant Laboratory or Applicant: the laboratory or organization applying for NELAP
- Assessment: the evaluation process used to measure or establish the performance, effectiveness, and conformance of an organization and/or its systems to defined criteria (to the standards and requirements of NELAC). (NELAC)

- Assessment (Clarification): The evaluation process used to measure the performance or effectiveness of a system and its elements against specific criteria.
- Assessment Criteria: the measures established by NELAC and applied in establishing the extent to which an applicant is in conformance with NELAC requirements. (NELAC)
- Assessment Team: the group of people authorized to perform the on-site inspection and proficiency testing data evaluation required to establish whether an applicant meets the criteria for NELAP accreditation. (NELAC)
- Assessor: one who performs on-site assessments of accrediting authorities and laboratories' capability and capacity for meeting NELAC requirements by examining the records and other physical evidence for each one of the tests for which accreditation has been requested. (NELAC)
- Audit: a systematic evaluation to determine the conformance to quantity tive and qualitative specifications of some operational function or activity. (EPA QAN)
- Batch: environmental samples, which are prepared a. '/or tralyzid together with the same process and personnel, using the same lot(s, of trage to. A preparation batch is composed of one to 20 environmental samples if the same NELAC-defined matrix, meeting the above mentioned criteria and train to next the start of processing of the first and last sample in the batch to be 24 hours. An analytical batch is composed of prepared environmental samples for tracts, digestates or concentrates), which are analyzed together as grow. An analytical batch can include prepared samples originating from various environmental matrices and can exceed 20 samples. (NELAC Quality Systems Commune 1)
- Blank: a sample that has not bee, exposed to the analyzed sample stream in order to monitor contamination uring sampling, transport, storage or analysis. The blank is subjected to the usual a alytic: and measurement process to establish a zero baseline or background value and is constitues used to adjust or correct routine analytical results. Blanks include:

**Equipment 3Ians** a sample of analyte-free media, which has been used to rinse common san. Jing equipment to check effectiveness of decontamination procedures. (NELAC)

Field Rap'. blank prepared in the field by filling a clean container with pure de-ionized water and appropriate preservative, if any, for the specific sampling activity being undertaken. (EPA OSWER)

**Instrument Blank:** a clean sample (e.g. distilled water) processed through the instrumental steps of the measurement process; used to determine instrument contamination. (EPA-QAD)

**Method Blank;** a sample of a matrix similar to the batch of associated samples (when available) that is free from the analytes of interest and is processed simultaneously with and under the same conditions as samples through all steps of the analytical procedures, and in which no target analytes or interferences are present at concentrations that impact the analytical results for sample analyses, (NELAC)

**Reagent Blank:** (method reagent blank): a sample consisting of reagent(s), without the target analyte or sample matrix, introduced into the analytical procedure at the appropriate point and carried through all subsequent steps to determine the contribution of the reagents and of the involved analytical steps. (QAMS)

- Blind Sample: a sub-sample for analysis with a composition known to the submitter. The analyst/laboratory may know the identity of the sample but not its composition. It is used to test the analyst or laboratory's proficiency in the execution of the measurement process. (NELAC)
- **Calibration:** set of operations which establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (VIM: 6.11)
  - In calibration of support equipment the values realized by standards are established through the use of Reference Standards that are traceable to the International System of Units (SI).
  - 2) In calibration according to test methods, the values real ad by standards are typically established through the use of Reference Vater, is that are either purchased by the Laboratory with a certificate of analysis or printy, or prepared by the Laboratory using support equipment that has peer calibrated verified to meet specifications.
- **Calibration Range:** The range of values (concentra, ons) between the lowest and highest calibration standards of a multi-level calibration decive. For metals analysis with a single-point calibration, the lowlevel calibration check standard and the high standard establish the linear calibration range, which lies within the linear calibration.
- Calibration Curve: the graphical relations of between the known values, such as concentrations, of a series of a vibrational standards and their instrument response. (NELAC)
- Calibration Method: a defir ed technical procedure for performing a calibration. (NELAC)
- Calibration Standard a subsence or reference material used to calibrate an instrument. (QAMS)
- Certified Pefe: nce h sterial (CRM): a reference material one or more of whose property values a signal contribution of the state of th
- Chain of Custody Form: record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; collector; time of collection; preservation; and requested analyses. (NELAC)
- **Clean Air Act:** the enabling legislation in 42 U.S.C. 7401 *et seq.*, Public Law 91-604, 84 Stat. 1676 Pub.L. 95-95, 91 Stat., 685 and Pub. L. 95-190, 91 Stat., 1399, as amended, empowering EPA to promulgate air quality standards, monitor and to enforce them. (NELAC)
- **Client:** Any individual or organization for whom items or services are furnished or work performed in response to defined requirements and expectations. (ANSI/ASQ E4-2004)

**Congener:** A member of a class of related chemical compounds (e.g., PCBs, PCDDs)

- Comprehensive Environmental Response, Compensation and Liability Act (CERCLA/Superfund): the enabling legislation in 42 U.S.C. 9601-9675 et seq., as amended by the Superfund Amendments and Reauthorization Act of 1986 (SARA), 42 U.S.C. 9601 et seq., to eliminate the health and environmental threats posed by hazardous waste sites. (NELAC)
- **Confidential Business Information (CBI):** information that an organization designates as having the potential of providing a competitor with inappropriate insight into its management, operation or products. NELAC and its representatives agree to safeguarding identified CBI and to maintain all information fue tified as such in full confidentiality.
- Confirmation: verification of the identity of a component through the use of an approach with a different scientific principle from the original method. These may include, but are not limited to:
  - Second column confirmation Alternate wavelength Dervitization Mass spectral interpretation Alternative detectors or Additional cleanup procedures. (NELAC)
- Conformance: an afrimative indication or judgment that a product or service has met the requirements on the Covant specifications, contract, or regulation; also the state of meeting the equirements. (ANSI/ASQC E4-1994)
- Cons nsus Sta dard: A standard established by a group representing a cross-section of a
  - partic. 'ar ir Justry or trade, or a part thereof. (ANSI/ASQ ANSI/ASQ E4-2004)

**Continuing calibration verification**: The verification of the initial calibration that is required during the course of analysis at periodic intervals. Continuing calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. (IDQTF)

**Contributor:** a participant in NELAC who is not a Voting Member. Contributors include representatives of laboratories, manufacturers, industry, business, consumers, academia, laboratory associations, laboratory accreditation associations, counties, municipalities, and other political subdivisions, other federal and state officials not engaged in environmental activities, and other persons who are interested in the objectives and activities of NELAC.

- **Corrective Action:** the action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)
- **Critical Finding:** a finding or a combination of findings that results in a significant negative effect on data quality or defensibility, if not corrected. (NELAC)
- **Data Audit:** a qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality (i.e., that they meet specified acceptance criteria.) (NELAC)

Data Reduction: the process of transforming raw data by arithmetic or statistical calculations, standard curves, concentration factors, etc., and collation into a more useable form. (EPA-QAD)

Deficiency: See Finding and Critical Finding

**Definitive Data:** Analytical data of known quality, concerdation, and level of uncertainty. The levels of quality and uncertainty f the solution of the solut

- Delegate: any environmental official of the Sta. s or the Federal government not sitting in the House of Representatives, who is slig ble to vote in the House of Delegates. (NELAC)
- **Demonstration of Capability:** a produce the establish the ability of the analyst to generate acceptable accuracy. (NELAL
- **Denial:** to refuse to accredi in to, 1 or in part a laboratory applying for initial accreditation or resubmission of initial application. (NELAC) [4.4.1]

**Detection Limit**: the lower concentration or amount of the target analyte that can be identified measured, and reported with confidence that the analyte conceleration is not a false positive value. See Method Detection Limit. (NEL,  $\Im$ )

Det. -tion Lim (DL) (Clarification): The smallest analyte concentration that can be 'emr' strated to be different from zero or a blank concentration at the 99% level of confidence. At the DL, the false positive rate (Type I error) is 1%.

**Document Control:** the act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly and controlled to ensure use of the correct version at the location where the prescribed activity is performed. (ASQC)

**Environmental Data:** Any measurements or information that describe environmental processes, locations, or conditions; ecological or health effects and consequences; or the performance of environmental technology. (ANSI/ASQ E4-2004)

Environmental Laboratory Advisory Board (ELAB): a Federal Advisory Committee, with members appointed by EPA and composed of a balance of non-state, non-federal representatives, from the environmental laboratory community, and chaired by an ELAB member. (NELAC)

Environmental Monitoring Management Council (EMMC): an EPA Committee consisting of EPA managers and scientists, organized into a Policy Council, a Steering Group, *ad hoc* Panels, and work groups addressing specific objectives, established to address EPAwide monitoring issues. (NELAC)

False Negative: An analyte incorrectly reported as absent from the sample, resulting in potential risks from their presence.

False Positive: An item incorrectly identified as present in the sample, resulting in a high reporting value for the analyte of concern.

- Federal Insecticide, Fungicide and Rodenticide Act (FIFPA). the enabling legislation under 7 U.S.C. 135 *et seq.*, as amended, that empower the *L*PA to register insecticides, fungicides, and rodenticides. (NELAC)
- Federal Water Pollution Control Act (Clean Water Act, GWA): the enabling legislation under 33 U.S.C 1251 et seq., Public Law 92-200-3 Sect. 8.16, that empowers EPA to set discharge limitations, write discharge permis, more tor, and bring enforcement action for non-compliance. (NELAC)
- Field Measurement: The determination f physical, biological, or radiological properties, or chemical constituents; that are rice sure in arsite, close in time and space to the matrices being sampled/measured, foll ving a cepted test methods. This testing is performed in the field outside of a fixed-labor tory or outside of an enclosed structure that meets the requirements of a mobile rabor tory.
- Field of Accreditatio .: (preciously Field of Testing) NELAC's approach to accrediting laboratories by matrix, schnology/method and analyte/analyte group. Laboratories requesting to sea, stick for a matrix-technology/method-analyte/analyte group combination or for an updated/improved method are required to submit only that portion of the accredition process not previously addressed. (NELAC)
- Field S Profici ncy Testing: NELAC's approach to offering proficiency testing by matrix, tech. logv and analyte/analyte group.
- Finding: an assessment conclusion, referenced to a NELAC Standard and supported by objective evidence that identifies a deviation from a NELAC requirement. See Critical Finding.

**Finding (Clarification):** An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding may be positive or negative and is normally accompanied by specific examples of the observed condition (ANSI/ASQ E4-2004).

**Governmental Laboratory:** as used in these standards, a laboratory owned by a Federal, state or tribal government; includes government-owned contractor-operated laboratories. (NELAC)

Holding Times (Maximum Allowable Holding Times): the maximum times that samples may be held prior to analysis and still be considered valid or not compromised. (40 CFR part 136)

Holding Times (DoD Clarification): The time elapsed from the time of sampling to the time of extraction or analysis, or from extraction to analysis, as appropriate.

- Inspection: an activity such as measuring, examining, testing, or gauging one or more characteristics of an entity and comparing the results with specified requirements in order to establish whether conformance is achieved for each characteristic. (ANSI/ASQC E4-1994)
- Interim Accreditation: temporary accreditation status for a looral ry that has met all accreditation criteria except for a pending on-site assessmer. which has been delayed for reasons beyond the control of the laboratory. (NELAC)
- Internal Standard: a known amount of standard dec to dec t
- **Isomer:** One of two or more compounds, rac cal. or ic is that contain the same number of atoms of the same elements but dif or in . ructural arrangement and properties. For example, hexane (C6H14) could be ... exal. 2-methylpentane, 3-methylpentane, 2,3-dimethylbutane, 2,2-dimethylbutae

Laboratory Control Sample (by wever named, such as laboratory fortified blank, spiked blank or QC heck sample): a sample matrix, free from the analytes of interest, spiked with verified income nounts of analytes or a material containing known and verified amounts of analytes. It is generally used to establish intra-laboratory or analyst specific precision a. 1 bits or to assess the performance of all or a portion of the measurement system. (NEL/ $\Im$ ).

- Laborat. v Dv licate: aliquots of a sample taken from the same container under laboratory conditions and processed and analyzed independently. (NELAC)
- Legal Chain of Custody Protocols: procedures employed to record the possession of samples from the time of sampling until analysis and are performed at the special request of the client. These protocols include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples by the laboratory. In addition, these protocols document all handling of the samples within the laboratory. (NELAC)
- Limit of Detection (LOD): An estimate of the minimum amount of a substance that an analytical process can reliably detect A LOD is analyte-and-matrix-specific and may be laboratory-dependent.

Limit of Detection (Clarification): The smallest amount or concentration of a substance that must be present in a sample in order to be detected at a high level of confidence (99%). At the LOD, the false negative rate (Type II error)

is 1%.

Limits of Quantitation (LOQ): The minimum levels, concentrations, or quantities of a target variable (e.g. target analyte) that can be reported with a specified degree of confidence.

Limit of Quantitation (Clarification): The lowest concentration that produces a quantitative result within specified limits of precision and bias. For DoD projects, the LOQ shall be set at or above the concentration of the lowest initial calibration standard.

Manager (however named): the individual designated as being i sponsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A supervisor may report to the manager. In some cases, the spervisor and the manager may be the same individual. (NELAC)

**Management:** Those individuals directly responsible of a countable for planning, implementing, and assessing work. (ANS. AStar 54-2004)

**Management System:** System to establish policy and objectives and to achieve those objectives (ISO 9000)

Matrix: the substrate of a test samp'.

Field of Accreditation Matrix: tr. se matrix definitions shall be used when accrediting a laboratory (see Field of / ccreu atiun).

- Drinking w .ter: An, ... yueous sample that has been designated a potable or potentia potable water source.
- Non- ouble state: Any aqueous sample excluded from the definition of a outling ater matrix. Includes surface water, groundwater, effluents, outer state themicals, and TCLP or other extracts.
- Sol, and Chemical Materials: includes soils, sediments, sludges, provucts and by-products of an industrial process that results in a matrix , previously defined.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers and the extracted concentrated analytes of interest from a gas or vapor that are collected with a sorbent tube, impinger solution, filter or other device. (NELAC)

Quality System Matrix: These matrix definitions are an expansion of the field of accreditation matrices and shall be used for purposes of batch and quality control requirements (see Appendix D of Chapter 5). These matrix distinctions shall be used:

• Aqueous: any aqueous sample excluded from the definition of Drinking Water matrix or Saline/Estuarine source. Includes surface water, groundwater, effluents, and TCLP or other extracts.

- Drinking water: Any aqueous sample that has been designated a potable or potential potable water source.
- Saline/Estuarine: Any aqueous sample from an ocean or estuary, or other salt water source such as the Great Salt Lake.
- Non-aqueous liquid: Any organic liquid with <15% settleable solids.
- Biological Tissue: Any sample of a biological origin such as fish tissue, shellfish, or plant material. Such samples shall be grouped according to origin.
- Solids: Includes soils, sediments, sludges and other matrices with >15%
  settleable solids.
- Chemical Waste: A product or by-product of an industrial process that results in a matrix not previously defined.
- Air and Emissions: whole gas or vapor samples including those contained in flexible or rigid wall containers end the extracted concentrated analytes of interest from a gas or vapor that end collected with a sorbent tube, impinger solution, filter or other. (av. (NELAC))
- Matrix Spike (spiked sample, fortified sample): a s.mp. prepared by adding a known mass of target analyte to a specified amount of <u>1.3trix</u> sample for which an independent estimate of Target analyte concentration is available. Matrix spikes are used, for example, to determine the effect of the natrix or a method's recovery efficiency. (QAMS).
- Matrix Spike Duplicate (spiked samper or nortified sample duplicate): a second replicate matrix spike prepared in the laboratory and analyzed to obtain a measure of the precision of the recovery for each analyte (Q, MS).
- May: denoted permitted actic ., L \* nu required action. (NELAC)
- Measurement Quality *iver* (MQOs): the desired sensitivity, range, precision, and bias of a measurement.
- Measurement S ste. ∵ a test method, as implemented at a particular laboratory, and which include the guipm int used to perform the test and the operator(s).
- Meth 1: 1. & e Test Method. 2. Logical sequence of operations, described generically, use in the enformance of measurements. (VIM 2.4)
- Method Detection Limit :one way to establish a Limit of Detection, defined as the minimum concentration of a substance (an analyte) that can be measured and reported with 99% confidence that the analyte concentration is greater than zero and is determined from analysis of a sample in a given matrix containing the analyte.

**Method Detection Limit (MDL) (Clarification):** The MDL is one way to establish a Detection Limit, not a Limit of Detection.

**Method of Standard Additions:** A set of procedures adding one or more increments of a standard solution to sample aliquots of the same size in order to overcome inherent matrix effects. The procedures encompass the extrapolation back to obtain the sample concentration. (This process is often called spiking the sample.) (Modified Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

**Mobile Laboratory**: A portable enclosed structure with necessary and appropriate accommodation and environmental conditions as described in Chapter 5, within which testing is performed by analysts. Examples include but are not limited to trailers, vans and skid-mounted structures configured to house testing equipment and personnel.

Must: denotes a requirement that must be met. (Random House College Dictionary)

- **National Accreditation Database:** the publicly accessible database listing the accreditation status of all laboratories participating in NELAP. (NELAC)
- National Institute of Standards and Technology (NIST): an age v of the US Department of Commerce's Technology Administration that is working with Lr. States, NELAC, and other public and commercial entities to establish a system nder hich private sector companies and interested States can be accredited by MIST. private NIST-traceable proficiency testing (PT) to those laboratories testing drining water and wastewater. (NIST)
- National Environmental Laboratory Accreditation `on, rence (NELAC): a voluntary organization of State and Federal environmenta, officials and interest groups purposed primarily to establish mutually accepted stan ards for accrediting environmental laboratories. A subset of NELAP. (NELAC)
- National Environmental Laboratory Acc. Jitation Program (NELAP): the overall National Environmental Laboratory Acc. Jitation Program of which NELAC is a part. (NELAC)
- National Voluntary Labora bry A creditation Program (NVLAP): a program administered by NIST that is used L providers of proficiency testing to gain accreditation for all compound/matrice for Winch NVLAP accreditation is available, and for which the provider intends to provid NELAP PT samples. (NELAC)
- **Negative Cont.** I: met oures taken to ensure that a test, its components, or the environment do not conserve the ordesired effects, or produce incorrect test results. (NELAC)
- NELAL Standa Js: the plan of procedures for consistently evaluating and documenting the ability flc oratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (NELAC)
- **NELAP Recognition:** the determination by the NELAP Director that an accrediting authority meets the requirements of the NELAP and is authorized to grant NELAP accreditation to laboratories. (NELAC)
- **Performance Audit:** the routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)
- **Positive Control:** measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (NELAC)
- Precision: the degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves; a data quality indicator.

Precision is usually expressed as standard deviation, variance or range, in either absolute or relative terms. (NELAC ).

- **Preservation**: refrigeration and/or reagents added at the time of sample collection (or later) to maintain the chemical and/or biological integrity of the sample. (NELAC)
- **Primary Accrediting Authority:** the agency or department designated at the Territory, State or Federal level as the recognized authority with responsibility and accountability for granting NELAC accreditation for a specified field of testing. (NELAC)
- **Procedure:** Specified way to carry out an activity or a process. Procedures can be documented or not. (ISO 9000: 2000 and Note 1)
- **Proficiency Testing:** a means of evaluating a laboratory's pertomance under controlled conditions relative to a given set of criteria through analy is of unknown samples provided by an external source. (NELAC) [2.1]
- Proficiency Testing Oversight Body/Proficiency estine Provider Accreditor (PTOB/PTPA): an organization with technical elipential, an inistrative capacity and financial resources sufficient to implement and organal and ional program of PT provider evaluation and oversight that meets the responsible field of requirements established by the NELAC Standards. (NELAC)
- Proficiency Testing Program: the age ega, or providing rigorously controlled and standardized environmental samp. r to plaboratory for analysis, reporting of results, statistical evaluation of the results and the collective demographics and results summary of all participating laboratories.
- Proficiency Testing Study Provide any person, private party, or government entity that meets stringent criteria o provide and distribute NELAC PT samples, evaluate study results against published performance criteria and report the results to the laboratories, primary accrediting authorities, PTOB/PTPA and NELAP. (NELAC)
- Proficiency Tes' ر 'm, ' , 'T): a sample, the composition of which is unknown to the analys' anc is pro ided to test whether the analyst/laboratory can produce analytical results v. 'hin pecified acceptance criteria . (QAMS).
- **Protoc !:** a det iled written procedure for field and/or laboratory operation (e.g., sampling, analy is) which must be strictly followed. (EPA-QAD)
- **Quality Assurance**: an integrated system of activities involving planning, quality control, quality assessment, reporting and quality improvement to ensure that a product or service meets defined standards of quality with a stated level of confidence. (QAMS,).
- Quality Assurance [Project] Plan (QAPP): a formal document describing the detailed quality control procedures by which the quality requirements defined for the data and decisions pertaining to a specific project are to be achieved. (EPA-QAD)
- **Quality Control**: the overall system of technical activities whose purpose is to measure and control the quality of a product or service so that it meets the needs of users. (QAMS).
- **Quality Control Sample**: a sample used to assess the performance of all or a portion of the measurement system. QC samples may be Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking.

**Quality Systems Manual:** A document stating the management policies, objectives, principles, organizational structure and authority, responsibilities, accountability, and implementation of an agency, organization, or laboratory, the ensure the quality of its product and the utility of its product to the users.

**Quantitation Range:** The range of values in a calibration curve between the LOQ and the highest successfully analyzed initial calibration standard. The quantitation range lies within the calibration range.

- Quality System: a structured and documented management system describing the policies, objectives, principles, organizational authority, responsibilities, accountability, and implementation plan of an organization for ensuring qualit, in its work processes, products (items), and services. The quality system provides the tramework for planning, implementing, and assessing work performed by the organitation and for carrying out required QA and QC. (ANSI/ASQC E-41994)
- Raw Data: any original factual information from a measure heat a tivity or study recorded in a laboratory notebook, worksheets, records, manuale notes, or exact copies thereof that are necessary for the reconstruction and evaluation of the report of the activity or study. Raw data may include photography for mor microfiche copies, computer printouts, magnetic media, including dictated or pervations, and recorded data from automated instruments. If exact copies of the vertice of the activity of the have been prepared (e.g., tapes which have been transcribed verbatime, the and verified accurate by signature), the exact copy or exact transcript may be sub if the copy of the table.
- Recognition: previously known as recipincity. The mutual agreement of two or more parties (i.e. States) to accept each oth it's findings regarding the ability of environmental testing laboratories in meeting Nillan standards. (NELAC)
- Reference Material: a .....en. ' or substance one or more properties of which are sufficiently well established t be u ed for the calibration of an apparatus, the assessment of a measurement meth. d. or for assigning values to materials. (ISO Guide 30 2.1)
- Reference `tan、 rd: a standard, generally of the highest metrological quality available at a given loca مرام which measurements made at that location are derived. (VIM 6.08)
- Reference Toy sant: the toxicant used in performing toxicity tests to indicate the sensitivity of a test organism and to demonstrate the laboratory's ability to perform the test correctly and obtain consistent results (see Chapter 5, Appendix D, Section 2.1f). (NELAC)
- **Replicate Analyses:** the measurements of the variable of interest performed identically on two or more sub-samples of the same sample within a short time interval. (NELAC)
- Requirement: Denotes a mandatory specification; often designated by the term "shall". (NELAC)
- Resource Conservation and Recovery Act (RCRA): the enabling legislation under 42 USC 321 *et seq*. (1976), that gives EPA the authority to control hazardous waste from the "cradle-to-grave", including its generation, transportation, treatment, storage and disposal. (NELAC)
- **Revocation:** the total or partial withdrawal of a laboratory's accreditation by the accrediting authority. (NELAC) [4.4.3]

- Safe Drinking Water Act (SDWA): the enabling legislation, 42 USC 300f *et seq.* (1974), (Public Law 93-523), that requires the EPA to protect the quality of drinking water in the U.S. by setting maximum allowable contaminant levels, monitoring, and enforcing violations.
- Sample Tracking: procedures employed to record the possession of the samples from the time of sampling until analysis, reporting and archiving. These procedures include the use of a Chain of Custody Form that documents the collection, transport, and receipt of compliance samples to the laboratory. In addition, access to the laboratory is limited and controlled to protect the integrity of the samples. (NELAC)
- Secondary Accrediting Authority: the Territorial, State or federal agency that grants NELAC accreditation to laboratories, based upon their accreditation by a NELAPrecognized Primary Accrediting Authority. See also Rt ognition and Primary Accrediting Authority. (NELCA)

Second source calibration verification (ICV): A standary obtained or prepared from a source

independent of the source of standards for  $x \ge 0$ , sal combration. Its concentration should be at or near the middle of the call, ration range. It is done after the initial calibration.

- Selectivity: (Analytical chemistry) the collab. V of a test method or instrument to respond to a target substance or constituent in the presence of non-target substances. (EPA-QAD)
- Sensitivity: the capability of a test sethod or instrument to discriminate between measurement responses ting different levels (e.g., concentrations) of a variable of interest. (NELAC)
- Shall: denotes a requirement that is mandatory whenever the criterion for conformance with the specification requires that there be no deviation. This does not prohibit the use of alternative a promote or methods for implementing the specification so long as the requirement s fulfill. d. (ANSI).

**Sig al to Noi e Ratio:** The signal carries information about the analyte, while noise is made up of entrance us information that is unwanted because it degrades the accuracy and precision or an analysis and also places a lower limit on the amount of analyte that can be detected. In most measurements, the average strength of the noise is constant and independent of the magnitude of the signal. Thus, the effect of noise on the relative error of a measurement becomes greater and greater as the quantity being measured (producing the signal) decreases in magnitude. (Skoog, Holler, and Nieman. Principles of Instrumental Analysis. 1998)

- **Should:** denotes a guideline or recommendation whenever noncompliance with the specification is permissible. (ANSI).
- Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (Glossary of Quality Assurance Terms, QAMS, 8/31/92).

- **Spike:** a known mass of target analyte added to a blank sample or sub-sample; used to determine recovery efficiency or for other quality control purposes. (NELAC)
- **Standard:** the document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of NELAC and meets the approval requirements of NELAC procedures and policies. (ASQC)
- Standard Operating Procedures (SOPs): a written document which details the method of an operation, analysis or action whose techniques and procedures are thoroughly prescribed and which is accepted as the method for performing certain routine or repetitive tasks. (QAMS)
- Standard Method: a test method issued by an organization generally recognized as competent to do so.
- Standardized Reference Material (SRM): a certified reference mate al produced by the U.S. National Institute of Standards and Technology or other equivalant organization and characterized for absolute content, independent of analytical method.
- Statistical Minimum Significant Difference (SMS⊾`: ti > n.....ium difference between the control and a test concentration that is statistically significant; a measure of test sensitivity or power. The power of a test der ence in , art of the number of replicates per concentration, the significance level selected, e.g. 0.05, and the type of statistical analysis. If the variability remains constant, the ensitivity of the test increases as the number of replicates is increased. ("LA")
- Supervisor (however named): the individual (s) designated as being responsible for a particular area or category of inentity analysis. This responsibility includes direct day-today supervision of technical en, loyees, supply and instrument adequacy and upkeep, quality assurance/quality control outles and ascertaining that technical employees have the required balance c education, training and experience to perform the required analyses. (NELAC)
- Surrogate: a subjective in properties that mimic the analyte of interest. It is unlikely to be found in environment samples and is added to them for quality control purposes. (QAMS).
- Suspe. sion: te nporary removal of a laboratory's accreditation for a defined period of time, which shall not exceed six months, to allow the laboratory time to correct deficiencies or area of non-compliance with the NELAC standards. (NELAC) [4.4.2]

**Target Analytes:** Analytes specifically named by a client (also called project-specific analytes). Technical Director: Individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

- **Technical Director**: individual(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC).
- **Technology**: a specific arrangement of analytical instruments, detection systems, and/or preparation techniques.
- Test: a technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process or service according to a specified procedure. The result of a test is normally

recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2 - 12.1, amended)

**Test Method**: an adoption of a scientific technique for performing a specific measurement, as documented in a laboratory SOP or as published by a recognized authority.

**Testing Laboratory**: laboratory that performs tests. (ISO/IEC Guide 2 - 12.4)

**Testing Sensitivity/Power**: the minimum significant difference (MSD) between the control and test concentration that is statistically significant. It is dependent on the number of replicates per concentration, the selected significance level, and the type of statistical analysis (see Chapter 5, Appendix D, Section 2.4.a). (NELAC)

- Tolerance Chart: A chart in which the plotted quality control data is psessed via a tolerance level (e.g. +/- 10% of a mean) based on the precision level in L<sub>5</sub> d acceptable to meet overall quality/data use requirements instead of a statistical acceptation e criteria (e.g. +/- 3 sigma). (applies to radiobioassay laboratories). (ANSI)
- Toxic Substances Control Act (TSCA): the enabling legislation in 15 USC 2601 et seq. (1976), the provides for testing, regulating, and schemical produced or imported into the United States for possible toxic endets, rior to commercial manufacture. (NELAC)
- **Traceability:** the property of a result of <u>messura</u>ment whereby it can be related to appropriate standards, generally in <u>mathematicational</u> or national standards, through an unbroken chain of comparisons. (VIM 6.12)

**Tuning:** A check and/or adjuement of instrument performance for mass spectrometry as required by the neurod.

- United States Enviroment. Protection Agency (EPA): the federal governmental agency with responsibility to projecting public health and safeguarding and improving the natural environment (i.e., e air, water and land) upon which human life depends. (US-EPA)
- Validation: Le confirmation by examination and provision of objective evidence that the poticular reluirements for a specific intended use are fulfilled.
- Verification. confirmation by examination and provision of evidence that specified requirements have been met. (NELAC)
- NOTE In connection with the management of measuring equipment, verification provides a means for checking that the deviations between values indicated by a measuring instrument and corresponding known values of a measured quantity are consistently smaller than the maximum allowable error defined in a standard, regulation or specification peculiar to the management of the measuring equipment.

The result of verification leads to a decision either to restore in service, to perform adjustments, or to repair, or to downgrade, or to declare obsolete. In all cases, it is required that a written trace of the verification performed shall be kept on the measuring instrument's individual record.

Voting Member: Officials in the employ of the Government of the United States, and the States, the Territories, the Possessions of the United States, or the District of Columbia

and who are actively engaged in environmental regulatory programs or accreditation of environmental laboratories. (NELAC)

- **Work Cell:** a well-defined group of analysts that together perform the method analysis. The members of the group and their specific functions within the work cell must be fully documented. (NELAC)
- **Working Range:** the difference between the Limit of Quantitation and the upper limit of measurement system calibration.

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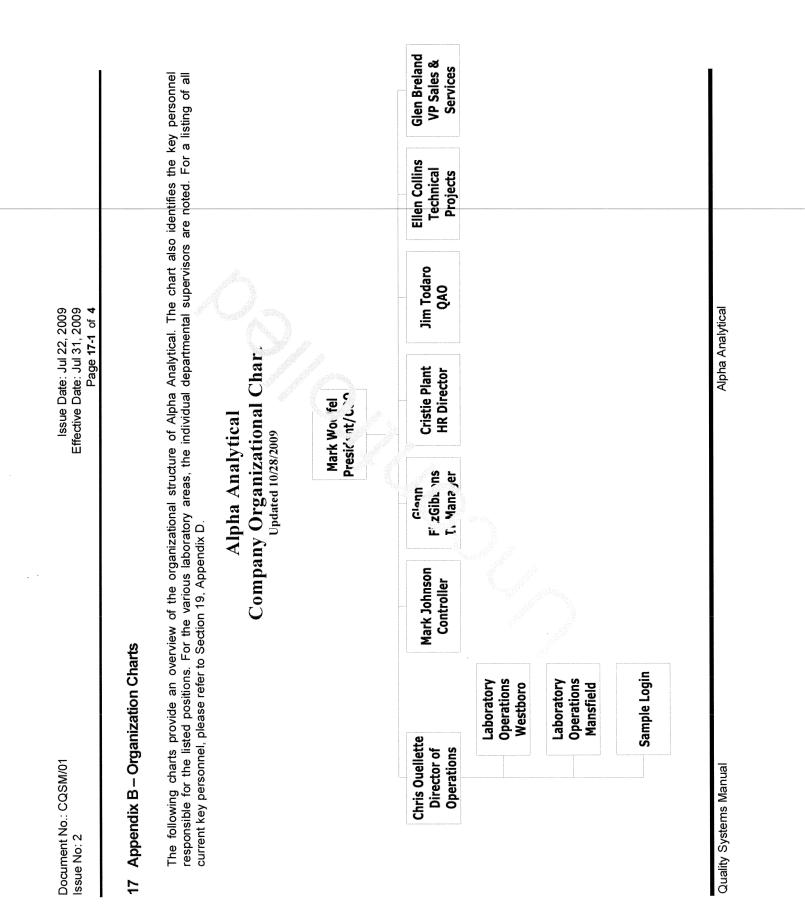
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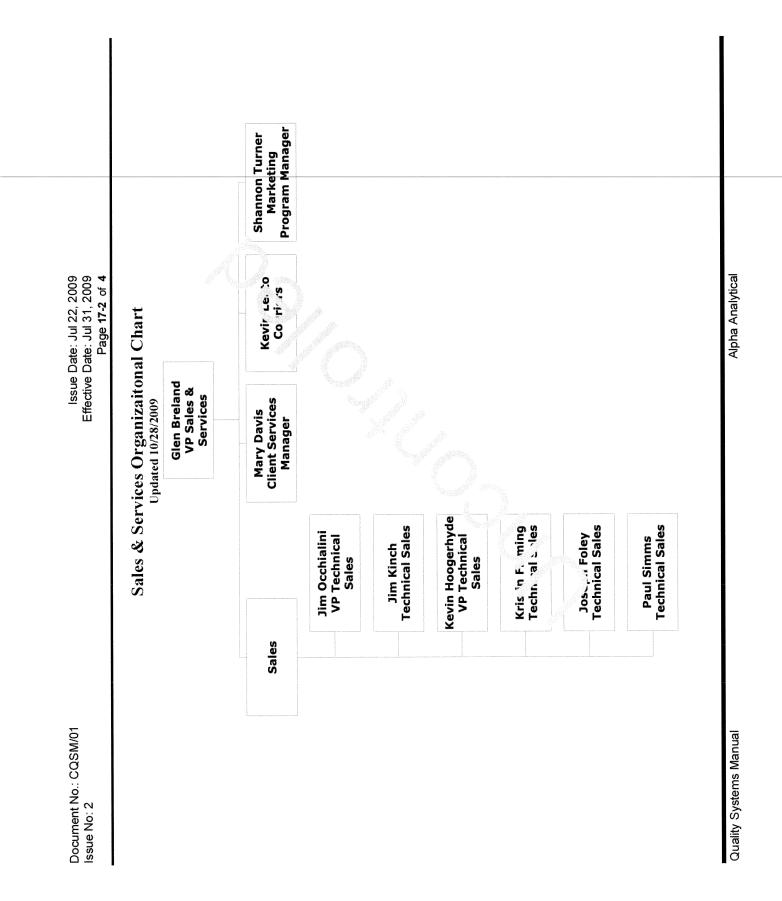
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e: Jul 31, 2009 Page 17-3 of 4	Laboratory Organizaitonal Chart WESTBOROUGH Updated 10/28/2009 Christopher Ouellette Director of Operations		≂lena Dayn Vet Chemistry / M∢tals Manager					Alpha Analytical
Issue Date: Jul 22, 2009 Effective Date: Jul 31, 2009 Page 17-3 of 4		Operations	Christopher Wakefield Lab/Technic .	Gardo. Tripp An 'ytical S) tems cingineer				٩
			Christopher Ouellette Interim ganics Manager	Jon Zygmuntowicz Organics Extractions Manager	Extracta <sup>J</sup> .e Organi s Ar ∴sis	Volat' e Analysis		
			Chri Ou Organi					
Issue No: 2			Lisa Westerlind Reporting &Login Manager	Kim Bailey Sample Login				Quality Systems Manual

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		Andy Rezendes Air & Volatile Manager				
Issue Date: Jul 22, 2009 Effective Date: Jul 31, 2009 Page 17-4 of 4	Alpha Analytical Laboratory Organizaitonal Chart MANSFIELD Updated 10/28/2009 Christopher Ouellette Director of Operations	Pete Henriksen S/T Product I ine Manager	Lenny Pitts Lab Director/Metals Manager	Wet Chemistry	Organic Extractions / Semi Volatiles Analysis	Alpha Analytical
CQSM/01		Liz Porta Forensics Manager				s Manual
Document No.: CQSM/01 Issue No: 2						Quality Systems Manual

### 18 Appendix C – List of Key Personnel

The following is a listing of all current key personnel. If role is specific to a facility it is denoted by either Westboro or Mansfield following the position title. **Updated 10/28/2009** 

President / Sales Manager: Mark Woelfel

Director of Operations: Christopher Ouellette

Laboratory Director / Technical Director, Westboro: Christopher Wakefield

Laboratory Director / Technical Director, Mansfield: Leonard Pitts

Quality Assurance Officer: James C. Todaro

Quality Systems Specialists: Amy Rice, Rene Ackerman

VP, Technical Projects: Ellen Collins

Human Resources Director: Cristie Plant

Vice Presidents, Technical Sales: Glen Breland, Ja. es Comaini

Technical Sales Reps: Jim Kinch, Kristin Flemi .g, ⊾ vin doogerhide; Paul Simms; Joe Foley

Controller: Mark Johnson

A/P, Purchasing: Jennifer Walters

Credit & Collections Supervisor: Holi, Gennas

Information Technology: G<sup>1</sup> Fit. ribbons

VP, Sales and Services: G on Bre and

Client Services Mana ver, W stboro: Mary Davis

Sediment/Tissu / Prc luct Line Manager, Mansfield: Peter Henriksen

Inorganics Libai ment Manager, Westboro: Elena Dayn

Organ. s Depa ment Supervisor, Westboro Christopher Ouellette, Interim

Organic Exactions Supervisor, Westboro: John Zygmuntowicz

Organic Technical Specialists: Scott Enright (Westboro), Cindy McQueen (Mansfield)

Volatiles Supervisor: Tim Reid

Air Technical Director/Volatiles Manager, Mansfield: Andy Rezendes

Forensics Manager, Mansfield: Liz Porta

Data Review, Reporting and Login Report Manager: Lisa Westerlind

Equipment Maintenance: Gordon Tripp

Environmental Health & Safety Coordinator: Jeanette Soucy

Courier Manager: Kevin Lento

Hazardous Materials Consultant: Triumvirate

### 19 Appendix D – Preventive Maintenance Procedures

Optimized Service-Calibration Intervals							
Equipment	Frequency	Type of Calibration or Maintenance					
Balances	semiannually daily	cleaning & operations check by service technician (external) calibration verification using Class S-1 certified weights					
COD Reactor	annually annually	complete operations check by service technician (external) reaction temperature verification					
Conductivity Bridge	annually each use	verification of cell constant complete operations check by service technician (external) calibration verification					
DI Water System	as needed monthly annually daily	complete operations check by service technician (external) Residual Chlorine check Biosuitability testing (external) pH and Conductivity check					
DO Meter	annually each use	complete operations check by serve techinian (external) calibration against air as specified by manufacturer					
Emergency/Safety Equipment	annually monthly daile	fire extinguishers and emergency ait inground check eye washes, showe, fire and at a d first aid kits checked					
Freezers Gas Chromatographs	daily as needed as needed beginning and end of batch and 10 to 20 samples as per method	temperature veri <sup>r</sup> ratio. injection port prepartion, learning of detectors initial multi-point calib. tion continuing calibration (CCV) against initial calibration					
ICP	Every other day Daily Annually Annually As needed	Chang rump					
Lachat analyzer	Daily As needed	Calib, tion, clean lines Change tubing, change O-rings					
Mass Spectrometers (GC & ICP)	bi-annually as needed 12 hour or d iny	change of mechanical pump oil by service technician (external) cleaning of source BFB, DFTPP or ICP-MS tune analysis followed by ICAL or CCV					
Mercury Analyzer	monthly each upp	clean cell and change pump windings calibration using multi-point curve					
Auto-pipettes	Mon ily Ari, ally	verification of accuracy verification of precision					
Microwave	Duar⊾⁴v A∵ually	power and temperature verification RPM verification					
Ovens	anr. ally dai'	complete operations check by service technician (external) temperature verification					
pH Meters		complete operations check by service technician (external) calibration using certified buffers					
Refrigerators (General Use)	daily	temperature verification					
Refrigerators (Sample Management)	daily	temperature verification					
Spectrophotometer	Semi-annually Semi-annually daily	cleaning & operations check by service technician (external) wavelength verification (external) continuing calibration verification (CCV) against initial calibration					
TCLP Rotator	annually	RPM verification					
Thermometers (Mercury/Alcohol)	annually	calibration against NIST traceable thermometer (internal)					
Thermometers (digital)	Quarterly	calibration against NIST traceable thermometer (external)					
Thermometer (NIST Traceable)	annually	calibration and certification of conformance (external)					
Turbidity meter	annually each use	cleaning & operations check by service technician (external) calibration using formazin					
Weights (Class S-1)	annually	service/calibration and certification of conformance (external)					

### 20 Appendix E – List of Analytical Methods

### **Certificate/Approval Program Summary**

Last revised July 7, 2009 - Westboro Facility

The following list includes only those analytes/methods for which certification/approval is currently held. For a complete listing of analytes for the referenced methods, please contact your Alpha Customer Service Representative.

# Connecticut Department of Public Health <u>Certificate/Lab ID</u>: PH-0574. NELAP Accredited Solid Waste/Soil.

*Drinking Water* (<u>Inorganic Parameters</u>: Color, pH, Turbidity, Conductivity, <u>Auslinity</u>, Chloride, Free Residual Chlorine, Fluoride, Calcium Hardness, Sulfate, Nitrate, Nitrite, Aluninum Antimony, Arsenic, Barium, Beryllium, Cadmium, Calcium, Chromium, Copper, Iron, Lead. <u>Agnosium</u>, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, <u>Abelan</u>, <u>Vanadium</u>, Zinc, Total Dissolved Solids, Total Organic Carbon, Total Cyanide, Perchlo<u>ste</u>, <u>Agnosium</u>, <u>Parameters</u>: <u>Haloacetic</u> Acids, Volatile Organics 524.2, Total Trihalomethanes 524.2, <u>1,2</u> Dible<u>2</u>-3-chloropropane (DBCP), Ethylene Dibromide (EDB).)

Wastewater/Non-Potable Water (Inorganic Parameters: Coo, pr Conductivity, Acidity, Alkalinity, Chloride, Total Residual Chlorine, Fluoride, Total Hardnes: Calci m Hardness, Silica, Sulfate, Sulfide, Ammonia, Kjeldahl Nitrogen, Nitrate, Nitrite, O-Phosp ate, Totri Phosphorus, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calsiu, Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Meniury, Aolybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Titrin, m, in adium, Zinc, Total Residue (Solids), Total Dissolved Solids, Total Suspended Solids (con-filterable), BOD, CBOD, COD, TOC, Total Cyanide, Phenolics, Foaming Agents (MBAS), Bron, le, Oil and Grease. <u>Organic Parameters</u>: PCBs, Organochlorine Pesticides, Technical Chlorenne, Toxaphene, 2,4-D, 2,4,5-T, 2,4,5-TP(Silvex), Acid Extractables (Phenols), Benzidines, Fithalat Esters, Nitrosamines, Nitroaromatics & Isophorone, Polynuclear Aromatic Hydrocarbonne, malocines, Collorinated Hydrocarbons, Volatile Organics.) *Solid Waste/Soil* (Inorganic Parameters Lead in Paint, pH, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Cicius Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thallium, Tin, Vanadium, Zinc, Reinde, Ignitability, Phenolics, Corrosivity, TCLP Leach (1311), Reactivity.

<u>Organic Parameters</u>: PCL Organochlorine Pesticides, Technical Chlordane, Toxaphene, Extractable Petroleum Hydroc rbons (LTPH), Dicamba, 2,4-D, 2,4,5-T, 2,4,5-TP(Silvex), Volatile Organics, Acid Extractables (Pheners), 3 2'-Dichlorobenzidine, Phthalates, Nitrosamines, Nitroaromatics & Cyclic Ketones, PAHs, Haloetness, Chlorinated Hydrocarbons.)

### Maine Department of Human Services Certificate/Lab ID: 2009024.

*Drinking Water* (Inorganic Parameters: SM9215B, 9221E, 9222B, 9222D, 9223B, EPA 150.1, 180.1, 300.0, 353.2, SM2130B, 2320B, 4500CI-D, 4500CN-C, 4500CN-E, 4500F-C, 4500H+B,4500NO3-F, EPA 200.7, EPA 200.8, 245.1. <u>Organic Parameters</u>: 504.1, 524.2, SM 6251B.) *Wastewater/Non-Potable Water* (Inorganic Parameters: EPA 120.1, 1664A, 350.1, 351.1, 353.2, 410.4, 420.1, Lachat 10-107-06-1-B, SM2320B, 2340B, 2510B, 2540C, 2540D, 426C, 4500CI-D, 4500CI-E, 4500CN-C, 4500CN-E, 4500CN-E, 4500F-B, 4500F-C, 4500H+B, 4500Norg-B, 4500Norg-C, 4500NH3-B, 4500H3-B, 4500H3-B, 4500H3-B, 4500H3-B, 4500H3-B, 4500H3-B, 4500H3-B, 4500H3-B, 450H3-B, 450H3-B, 450H3-B,

G, 4500NH3-H, 4500NO3-F, 4500P-B.5, 4500P-E, 5210B, 5220D, 5310C, EPA 200.7, 200.8, 245.1. <u>Organic Parameters</u>: 608, 624.)

### Massachusetts Department of Environmental Protection Certificate/Lab ID: M-MA086.

Drinking Water

Inorganic Parameters: (EPA 200.8 for: Sb,As,Ba,Be,Cd,Cr,Cu,Pb,Ni,Se,Tl) (EPA 200.7 for: Ba,Be,Ca,Cd,Cr,Cu,Na,Ni) 245.1, (300.0 for: Nitrate-N, Nitrite-N, Fluoride, Sulfate) 353.2 for: Nitrate-N, Nitrite-N; SM4500NO3-F, 4500F-C, 4500CN-CE, EPA 180.1, SM2130B, SM4500CI-D, 2320B, SM2540C, EPA 150.1, SM4500H-B. <u>Organic Parameters</u>: (EPA 524.2 for: Trihalomethanes, Volatile Organics) (504.1 for: 1,2-Dibromoethane, 1,2-Dibromo-3-Chloropropane), SM6251B, 314.0.

### Non-Potable Water

Inorganic Parameters:, (EPA 200.8 for: Al,Sb,As,Be,Cd,Cr,Cu,Pb,Mn,Ni,Se,Ag,Tl,Zn) (EPA 200.7 for: Al,Sb,As,Be,Cd,Cr,Co,Cu,Fe,Pb,Mn,Mo,Ni,Se,Ag,Sr,Tl,Ti,V,Zn,Ca,Mg,Na,K) 245.1, SM4500H,B, EPA 120.1, SM2510B, 2540C, 2540B, 2320B, 4500CL-E, 4500F-BC, 426C, SM4500NH3-BH, (EPA 350.1 for: Ammonia-N), LACHAT 10-107-06-1-B for Nicote-N, SM4500NO3-F, 353.2 for Nitrate-N, SM4500NH3-B,C-Titr, SM4500NH3-BC-NES, EPA 351.1, SM 500P-E, 4500P-B,E, 5220D, EPA 410.4, SM 5210B, 5310C, 4500CN-CE, 2540D, 4500CL-D, EPA 364, M14 510AC, EPA 420.1

<u>Organic Parameters</u>: (EPA 624 for Volatile Halocarbons, Volatile Aromatius) (608 for: Chlordane, Aldrin, Dieldrin, DDD, DDE, DDT, Heptachlor, 'Hep and Jor Spoxide, PCB-Water) 600/4-81-045-PCB-Oil

Massachusetts Department of Environmental Protect on <u>`ertifir ate/Lab ID</u>: M-MA086. Drinking Water

Microbiology Parameters: SM9215B; MF-SM9222E, . NZ. UB. SM9223; EC-SM9221E; MF-SM9222D; ENZ. SUB. SM9223;

## New Hampshire Department of Environ, ental Services <u>Certificate/Lab ID</u>: 200307. NELAP Accredited.

*Drinking Water* (Inorganic Parameters: SM62 5B, 9222B, 9223B Colilert, EPA 200.7, 200.8, 245.2, 110.2, 120.1, 150.1, 300.0, 325.2, ^...0, <sup>SM/</sup> 00CN-E, 4500H+B, 4500NO3-F, 2320B, 2510B, 2540C, 4500F-C, 5310C, 2120B, EPA 331 ). <u>Org nic Parameters</u>: 504.1, 524.2, SM6251B.)

Non-Potable Water (Inorganic Pal. metr. s: SM9222D, 9221B, 9222B, 9221E-EC, EPA 200.7, 200.8, 245.1, 245.2, SW-846 6010 , 6 0, 7196A, 7470A, SM3500-CR-D, EPA 120.1, 150.1, 300.0, 305.1, 310.1, 325.2, 340.2, 3 1, 50.2, 51.1, 353.2, 354.1, 365.2, 375.4, 376.2, 405.1, 415.1, 420.1, 425.1, 1664A, SW-846 9010, 5.30, 040B, EPA 160.1, 160.2, 160.3, SM426C, SM2310B, 2540B, 2540D, 4500H+B, 4500N. 3-H, 4500 NH3-E, 4500NO2-B, 4500P-E, 4500-S2-D, 5210B, 2320B, 2540C, 4500F-C, 5310C, 5540C, LAC 'AT 10 17-07-1-B, LACHAT 10-107-06-1-B, LACHAT 10-107-04-1-C, LACHAT 10-107-04-1-J, LACHAT ... 17-07-1-A, SM4500CL-E, LACHAT 10-204-00-1-A, LACHAT 10-107-06-2-D. Organic Parameters: SW-846 3005A, 3015A, 3510C, 5030B, 8021B, 8260B, 8270C, 8330, EPA 624, 625, 608, SW-846 8082, 8081A.)

*Solid & Chemical Materials* (<u>Inorganic Parameters</u>: SW-846 6010B, 7196A, 7471A, 7.3.3.2, 7.3.4.2, 1010, 1030, 9010, 9012A, 9014, 9030B, 9040, 9045C, 9050C, 1311, 3005A, 3050B, 3051A. <u>Organic Parameters</u>: SW-846 3540C, 3545, 3580A, 5030B, 5035, 8021B, 8260B, 8270C, 8330, 8151A, 8082, 8081A.)

New Jersey Department of Environmental Protection <u>Certificate/Lab ID</u>: MA935. *NELAP Accredited. Drinking Water* (Inorganic Parameters: SM9222B, 9221E, 9223B, 9215B, 4500NO3-F, 4500F-C, EPA 300.0, 200.7, 2540C, 2320B, 314.0, 331.0, 110.2, SM2120B, 2510B, 5310C, EPA 150.1, SM4500H-B, EPA 200.8, 245.2. <u>Organic Parameters</u>: 504.1, SM6251B, 524.2.)

Non-Potable Water (Inorganic Parameters: SM5210B, EPA 410.1, SM5220D, 4500CI-D, EPA 300.0, SM2120B, SM4500F-BC, EPA 200.7, 351.1, LACHAT 10-107-06-2-D, EPA 353.2, SM4500NO3-F, 4500NO2-B, EPA 1664A, SM5310B, C or D, 4500-PE, EPA 420.1, SM4500P-B5+E, 2540B, 2540C, 2540D, EPA 120.1, SM2510B, SM15 426C, SM9221CE, 9222D, 9221B, 9222B, 9215B, 2310B, 2320B, 4500NH3-H, 4500-S D, EPA 350.2/.1, SM5210B, SW-846 3015, 6020, 7470A, 5540C, 4500H-B, EPA

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200.8, SM3500Cr-D, EPA 245.1, 245.2, SW-846 9040B, 3005A, EPA 6010B, 7196A, SW-846 9010B, 9030B. <u>Organic Parameters</u>: SW-846 8260B, 8270C, 3510C, EPA 608, 624, 625, SW-846 5030B, 8021B, 8081A, 8082, 8151A, 8330, NJ OQA-QAM-025 Rev.7.)

*Solid & Chemical Materials* (<u>Inorganic Parameters</u>: SW-846 9040B, 3005A, 6010B, 7196A, 5030B, 9010B, 9030B, 1030, 1311, 3050B, 3051, 7471A, 9014, 9012A, 9045C, 9050A, 9065. <u>Organic Parameters</u>: SW-846 8021B, 8081A, 8082, 8151A, 8330, 8260B, 8270C, 1311, 1312, 3540C, 3545, 3550B, 3580A, 5035L, 5035H, NJ OQA-QAM-025 Rev.7.)

### New York Department of Health Certificate/Lab ID: 11148. NELAP Accredited.

Drinking Water (Inorganic Parameters: SM9223B, 9222B, 8215B, EPA 200.8, 200.7, 245.2, SM5310C, EPA 314.0, 331.0, SM2320B, EPA 300.0, 325.2, 110.2, SM2120B, 4500CN-E, 4500F-C, EPA 150.1, SM4500H-B, 4500NO3-F, 2540C, EPA 120.1, SM 2510B. Organic Parameters: EPA 524.2, 504.1.) Non-Potable Water (Inorganic Parameters: SM9221E, 9222D, 9221B, 9222B, 9215B, EPA 405.1, SM5210B, EPA 410.4, SM5220D, EPA 305.1, SM2310B-4a, EPA 310.1, SM. 320B, EPA 200.7, 300.0, 325.2, LACHAT 10-117-07-1A or B, SM4500CI-E, EPA 340.2, SM4500F-C, EPA 75.4, SM15 426C, EPA 350.1, 350.2, LACHAT 10-107-06-1-B, SM4500NH3-H, EPA 351.1, LACHA 10-17-06-2, EPA 353.2, LACHAT 10-107-041-C, SM4500-NO30F, EPA 354.1, SM4500-NO2-B, EPA 365.2, SM4500P-E, EPA 160.3, EPA 160.1, SM2540C, EPA 160.2, SM2540B, SM2540D, EPA 201 3, L. A 6010B, 6020, EPA 7196A, S\M3500Cr-D, EPA 245.1, 245.2, 7470A, 110.2, SM2120L 33{ 2 LAC HAT 10-204-00-1-A, EPA 150.1, 9040B, SM4500-HB, EPA 1664A, EPA 415.1, SM5316 E. A . O. A. SM14 510C, EPA 120.1, SM2510B, EPA 376.2, SM4500S-D, EPA 425.1, SM5540C, EPA 3015. Organic Parameters: EPA 624, 8260B, 8270C, 625, 608, 8081A, 8151A, 8330, 8082, 800 E 4 3510C, 5030B, 9010B, 9030B.) Solid & Hazardous Waste (Inorganic Parameters: EPA 90/ JB, 9, 150, 1010, 1030, SW-846 Ch 7 Sec 7.3, EPA 6010B, 7196A, 7471A, 9012A, 9014, 9040B, J4 C, 9(35, 9050, EPA 1311, 3005A, 3050B, 9010B. 9030B. Organic Parameters: EPA 8260B 827C., 806TA, 8151A, 8330, 8082, 8021B, 3540C, 3545, 3580, 5030B, 5035.)

## Pennsylvania Department of Environmental rote tion <u>Certificate/Lab ID</u>: 68-03671. NELAP Accredited.

*Non-Potable Water* (<u>Organic Parameters</u>: \_\_, 35 °C, 625, 608, 8081A, 8082, 8151A, 8270C, 8330) *Solid & Hazardous Waste* (<u>Inorganic F</u> <u>rame</u> <u>rs</u>: EPA 1010, 1030, 1311, 3050B, 3051, 6010B, EPA 7.3.3.2, EPA 7.3.4.2, 7196A, 7471, s 10P, 9012A, 9014, 9040B, 9045C, 9050, 9065. <u>Organic</u> <u>Parameters</u>: 3540C, 3545, 358 A, 5°35, 8021B, 8081A, 8082, 8151A, 8260B, 8270C, 8330)

Rhode Island Department c. He. 'th <u>certificate/Lab ID</u>: LAO00065. *NELAP Accredited via NY-DOH.* Refer to MA-DEP Certilities or Poteble and Non-Potable Water. Refer to NY-DOH Certifice of for Potable and Non-Potable Water.

Utah Department of 1. <u>Certificate/Lab ID</u>: AAMA. *NELAP Accredited. Non-Potable Water* (<u>Inorganic Parameters</u>: Chloride EPA 300.0)

> Certificate/Approval Program Summary Last revised June 17, 2009 – Mansfield Facility

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The following list includes only those analytes/methods for which certification/approval is currently held. For a complete listing of analytes for the referenced methods, please contact your Alpha Customer Service Representative.

### Connecticut Department of Public Health Certificate/Lab ID: PH-0141.

*Wastewater/Non-Potable Water* (Inorganic Parameters: pH, Turbidity, Conductivity, Alkalinity, Aluminum, Antimony, Arsenic, Barium, Beryllium, Boron, Cadmium, Calcium, Chromium, Cobalt, Copper, Iron, Lead, Magnesium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Strontium, Thallium, Tin, Vanadium, Zinc, Total Residue (Solids), Total Suspended Solids (non-filterable), Total Cyanide. <u>Organic Parameters</u>: PCBs, Organochlorine Pesticides, Technical Chlordane, Toxaphene, Acid Extractables, Benzidines, Phthalate Esters, Nitrosamines, Nitroaromatics & Isophorone, PAHs, Haloethers, Chlorinated Hydrocarbons, Volatile Organics.)

Solid Waste/Soil (Inorganic Parameters: pH, Aluminum, Antimony, Arsenic, Ban, m, Beryllium, Cadmium, Calcium, Chromium, Hexavalent Chromium, Cobalt, Copper, Iron, Lead, Mag esium, Manganese, Mercury, Molybdenum, Nickel, Potassium, Selenium, Silver, Sodium, Thalli, m, Ve adium, Zinc, Total Organic Carbon, Total Cyanide, Corrosivity, TCLP 1311. <u>Organic Parameters</u> CBs, Organochlorine Pesticides, Technical Chlordane, Toxaphene, Volatile Organics, Aci. Txtractables, Benzidines, Phthalates, Nitrosamines, Nitroaromatics & Cyclic Ketches, F. Hs, Haloethers, Chlorinated Hydrocarbons.)

Florida Department of Health Certificate/Lab ID: E87814. Nr., ? A predited.

*Non-Potable Water* (<u>Inorganic Parameters</u>: SM2320B, 4<sup>-</sup>OL NH3-F EPA 120.1, SM2510B, 2340B, EPA 245.1, EPA 150.1, EPA 160.2, SM2540D, EPA 3(...2, -, SM2540G, EPA 180.1. <u>Organic Parameters</u>: EPA 625, 608.)

Solid & Chemical Materials (Inorganic Parametric 602 7470, 7471, 9045, 9014. Organic Parameters: EPA 8260, 8270, 8082, 8081.)

Air & Emissions (EPA TO-15.)

Louisiana Department of Environmental Quality Certificate/Lab ID: 03090. NELAP Accredited.

*Non-Potable Water* (<u>Inorganic Par meter</u>): ⊨PA 120.1, 150.1, 160.2, 180.1, 200.8, 245.1, 310.1, 335.2, 608, 625, 1631, 3010, 3015, 3020, 3020, 3010, 9014, 9040, SM2320B, 2510B, 2540D, 2540G, 4500CN-E, 4500H-B, <u>Organic Paraginete</u> 3: ⊨, A 3510, 3580, 3630, 3640, 3660, 3665, 5030, 8015 (mod), 3570, 8081, 8082, 8260, 827 )

Solid & Chemical Materia. (<u>Ir. rganic Parameters</u>: 6020, 7196, 7470, 7471, 7474, 9010, 9014, 9040, 9045, 9060. <u>Orc. nic Para leters</u>: EPA 8015 (mod), EPA 3570, 1311, 3050, 3051, 3060, 3580, 3640, 3660, 3665, 5u 5, 8<sup>o</sup> 1, 8082, 8260, 8270.)

*Biological Tissue* (<u>Inorganic Parameters</u>: EPA 6020. <u>Organic Parameters</u>: EPA 3570, 3510, 3610, 3630, 3640, 8270.)

Maine Department of Human Services Certificate/Lab ID: MA0030.

Wastewater (Inorganic Parameters: EPA 120.1, 300.0, SM 2320, 2510B, 2540C, 2540D, EPA 245.1. Organic Parameters: 608, 624.)

Massachusetts Department of Environmental Protection Certificate/Lab ID: M-MA030.

Non-Potable Water (Inorganic Parameters: SM4500H+B. Organic Parameters: EPA 624.)

New Hampshire Department of Environmental Services Certificate/Lab ID: 2206. NELAP Accredited.

*Non-Potable Water* (<u>Inorganic Parameters</u>: EPA 200.8, 245.1, 1631E, 120.1, 150.1, 180.1, 310.1, 335.2, 160.2, SM2540D, 2540G, 4500CN-E, 4500H+B, 2320B, 2510B. <u>Organic Parameters</u>: EPA 625, 608.)

### New Jersey Department of Environmental Protection Certificate/Lab ID: MA015. NELAP Accredited.

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*Non-Potable Water* (<u>Inorganic Parameters</u>: SW-846 1312, 3010, 3020A, 3015, 6020, SM2320B, EPA 200.8, SM2540C, 2540D, 2540G, EPA 120.1, SM2510B, EPA 180.1, 245.1, 1631E, SW-846 9040B, 6020, 9010B, 9014 <u>Organic Parameters</u>: EPA 608, 625, SW-846 3510C, 3580A, 5030B, 3035L, 5035H, 3630C, 3640A, 3660B, 3665A, 8081A, 8082 8260B, 8270C)

*Solid & Chemical Materials* (<u>Inorganic Parameters</u>: SW-846 6020, 9010B, 9014, 1311, 1312, 3050B, 3051, 3060A, 7196A, 7470A, 7471A, 9045C, 9060. <u>Organic Parameters</u>: SW-846 3580A, 5030B, 3035L, 5035H, 3630C, 3640A, 3660B, 3665A, 8081A, 8082, 8260B, 8270C, 3570, 8015B.)

Atmospheric Organic Parameters (EPA TO-15)

*Biological Tissue* (<u>Inorganic Parameters:</u> SW-846 6020 <u>Organic Parameters</u>: SW-846 8270C, 3510C, 3570, 3610B, 3630C, 3640A)

New York Department of Health Certificate/Lab ID: 11627. NELAP Accredite

*Non-Potable Water* (<u>Inorganic Parameters</u>: EPA 310.1, SM2320B, EPA <u>55.2</u>, 160.1, EPA 160.2, SM2540D, EPA 200.8, 6020, 1631E, 245.1, 335.2, 9014, 150.1, 9040B, 1201, SN 510B, EPA 376.2, 180.1, 9010B. <u>Organic Parameters</u>: EPA 624, 8260B, 8270C, 608, 850.1 CT 8082, 3510C, 3511, 5030B.)

Air & Emissions (EPA TO-15.)

Pennsylvania Department of Environmental <u>rotestion</u> <u>Certificate/Lab ID</u>: 68-02089. NELAP Accredited.

Non-Potable Water (Organic Parameters: EPA '036.) EFA 8260)

Rhode Island Department of Health Certificate, ab ID: LAO00299. NELAP Accredited via LA-DEQ.

Refer to MA-DEP Certificate for Non-Pot ble W ter.

Refer to LA-DEQ Certificate for Nor -Potabic ', ater.

Texas Commission of Environment I Quality Certificate/Lab ID: T104704419-08-TX. NELAP Accredited.

Solid & Chemical Mater. 's <u>corganic Parameters</u>: EPA 6020, 7471. <u>Organic Parameters</u>: EPA 8015, 8270.)

U.S. Army Corps o. Engingers

### 21 Appendix F – Alpha Code of Ethics Agreement

### Alpha Analytical, Inc. *Ethical Conduct and Data Integrity Agreement*

A. <u>Personal Pledge:</u> I understand that I am charged with meeting the highest degree of ethical standards in performing all of my duties and responsibilities and pledge to only report data, test results and conclusions that are accurate, precise and of the highest quality.

- B. <u>Protocol Pledges:</u> I agree to adhere to the following protocols and pr. ciples of ethical conduct in fulfilling my work assignments at Alpha:
  - 1. All work assigned to me will be performed using Standard Obertine Procedures (SOPs) that are based on EPA approved methods or Alpha met' ods
  - 2. I will only report results or data that match the ac value successful or measured.
  - 3. I will not intentionally nor improperly manipulate or to sify data in any manner, including both sample and QC data. Furthermore, I will not modify data values unless the modification can be technically justified the under a measurable analytical process or method acceptable to Alpha. All such modifications will be clearly and thoroughly documented in the appropriate labora pry rulebooks and raw data and include my initials or signature and date.
  - 4. I will not intentionally report date and times of analyses that are not the actual dates and times the analyses were conducted.
  - 5. I will not intention up rep. a lit another individual's work as my own or represent my work as someone. Ise's.
  - 6. I will not make failed statements to, or seek to otherwise deceive Alpha staff, leaders or clients. I will not, through acts of commission, omission, erasure or destruction, improperly apply measurements, standards results, data, test results or conclusions.

### C. <u>Guardian Aledge:</u>

- 1. I will not condone any accidental or intentional reporting of unauthentic data by other Alpha staff and will immediately report such occurrences to my supervisor, the QA Officer, the Laboratory Director or corporate leadership. I understand that failure to report such occurrences may subject me to immediate discipline, including termination.
  - 2. If a supervisor or other member of the Alpha leadership group requests me to engage in, or perform an activity that I feel is compromising data validity or quality, I have the right to not comply with the request and appeal this action through Alpha's QA Officer, senior leadership or corporate officers, including the President of the company.
  - 3. I understand that, if my job includes supervisory responsibilities, then I will not instruct, request or direct any subordinate to perform any laboratory practice that is unethical or improper. Also, I will not discourage, intimidate or inhibit a staff member who may

choose to appropriately appeal my supervisory instruction, request or directive that may be perceived to be improper, nor retaliate against those who do so.

D. <u>Agreement Signature:</u> I have read and fully understand all provisions of the Alpha Analytical Ethical Conduct and Data Integrity Agreement. I further realize and acknowledge my responsibility as an Alpha staff member to follow these standards. I clearly understand that adherence to these standards is a requirement of continued employment at Alpha.

Employee Signature

Printed Name

Date

#### **Review Requirements**

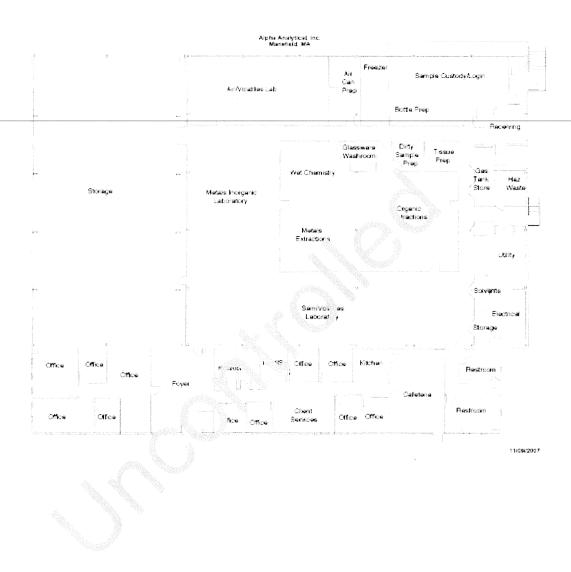
The Ethical Conduct and Data Integrity Agreement n ist be signed at the time of hire (or within 2 weeks of a staff member's receipt in the policy). Furthermore, each staff member will be required to review and sign this acreement every year. Such signature is a condition of continued employment at Alpha. Faure to comply with these requirements will result in immediate discharge from Alpha coology, ent. This agreement is not an employment contract and does not

Quality Systems Manual

## 22 Appendix G – Floor Plan Westboro Facility



Quality Systems Manual



# Appendix H– Floor Plan Mansfield Facility

Alpha Analytical, Inc.
Technical Standard Operating Procedure
Air Analysis; TO-15
Effective Date: May 22, 2009

Procedure No. SOP/A-001 Page 1 of 44 Issue No.:5 Rev 1 Issue Date:April 22, 2009

# Determination of Volatile Organic Compounds in Ambient Air Using Specially-Prepared Canisters and Analyzed by GC/MS

References: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition. U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Clincinnati, Ohio. January 1999.

Method TO-15: Determination of Volatile Organic Compounds (VOCs) in Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

Copy No.: \_\_\_\_\_

**Uncontrolled Document** 

Prepared By:

Name:	Andrew Rezendes	01	Position:	Product Line Manager-Air Testing
Signature:	Lil	KC.	Date:	4-22.09

Authorized By:

Name: Leonard C. Pitts, PhD Ho Ph.D. Signature

Position: Laboratory/Technical Director

Date: 4/22/09

#### **ISSUE AMENDMENTS**

Changes since last issue:

Reformatted SOP.

Section 2.1: Added method modifications for %RSD and for humidified nitrogen. Section 9.6: Added rotation of duplicate sample among clients/sites. Table 9A: Changed reporting limit for Methylene Chloride.

Removed Figures 1, 2, 4 and 5.

01/30/2009

# Determination of Volatile Organic Compounds in Ambient Air Using Specially-Prepared Canisters and Analyzed by GC/MS

References: Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air - Second Edition. U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

Method TO-15: Determination of Volatile Organic Compounds (VOCs) In Air Collected In Specially-Prepared Canisters and Analyzed By Gas Chromatography Mass Spectrometry (GC/MS). U.S. Environmental Protection Agency. EPA/625/R-96/010b. Office of Research and Development National Risk Management Research Laboratory. Center for Environmental Research Information. Cincinnati, Ohio. January 1999.

# 1. Scope and Application

#### Matrices: Air

Definitions: Refer to Section 16 and Alpha Analytical Quality Systems Manual

This SOP describes the procedure for the analysis of volatile organic compounds (VOCS) in ambient air. The whole air samples are collected in fused-silica lined (FSL) stainless steel canisters, or Tedlar® bags. The VOCs are subsequently separated by gas chromatography (GC) and measured by mass selective detector (MSD).

The organic compounds that are amenable to this method are listed in Table 9. Other compounds may also be amenable provided they meet the QA/QC requirements of the method.

The data report packages present the documentation of any method modification related to the samples tested. Depending upon the nature of the modification and the extent of intended use, the laboratory may be required to demonstrate that the modifications will produce equivalent results for the matrix. Approval of all method modifications is by one or more of the following laboratory personnel before performing the modification: Area Supervisor, Department Supervisor, Laboratory Director, or Quality Assurance Officer.

This method is restricted to use by or under the supervision of analysts experienced in the operation of the GC/MS and in the interpretation of GC/MS data. Each analyst must demonstrate the ability to generate acceptable results with this method by performing an initial demonstration of capability, analyzing a proficiency test sample and completing the record of training.

After initial demonstration, ongoing demonstration is based on acceptable laboratory performance of at least a quarterly laboratory control sample or acceptable performance from an annual proficiency test sample. A major modification to this procedure requires demonstration of performance. The identification of major method modification requiring performance demonstration is directed by the Quality Assurance Officer and/or Laboratory Director on a case-by-case basis.

# 2. Summary of Method

Samples are collected in precleaned, evacuated FSL canisters or Tedlar® bags.

Samples are preconcentrated using the Entech 7100A Cryogenic Concentrator. A specified volume of sample is pulled using a vacuum pump through a mass flow controller. The sample is cryogenically concentrated to a volume of less than one mL in a glass bead trap.

Following preconcentration, the sample is transferred to a Tenax® trap and then refocused on the GC transfer line. This step further reduces the sample volume to less than one microliter for injection.

The sample is then injected into the GC, which is used to separate the compounds of interest. All compounds are detected using an MSD.

## 2.1 Method Modifications from Reference

Initial Calibration modifications: If a target analyte can not meet the %RSD criteria for relative response factor calibration, then linear regression may be used. A minimum of five calibration points must be incorporated and a correlation coefficient of 0.995 or greater must be achieved. The calibration plot must be printed and approval by a supervisor must be obtained prior to calibration acceptance. If any compound is calibrated using linear regression then after the ICV and prior to any sample analysis, a low point standard must be analyzed to confirm there is no bias resulting from the linear regression calibration used. Recovery of the low point standard must be 60-140% using the linear regression curve.

Continuing calibration and laboratory controlled spike (LCS) modifications: The recoveries of all analytes must be within 70% to 130% of the true value. If more then 10% of the compounds fail these criteria, or if one compound has a recovery less than 50% or greater than 150% the LCS must be re-analyzed. If failure occurs a second time, the instrument must be re-calibrated.

Sample Duplicate modifications: Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte detected in one of the analysis at >5x the reporting limit, and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analysis, the RPD is NC.

Section 8.4.1.2 of the TO-15 method requires all canisters to be leak checked for a period of 24 hr. The laboratory conducts the leak check by measuring the vacuum of the canister over a 24 hr. period, not by pressurizing the canister as per the method.

The % RSD for any analyte must be < 30%, as outlined in Section 10.2.2.7 of this SOP.

Humidified nitrogen is used in place of zero air due to the frequency of detection of VOCs in zero air, particularly at SIM detection limits.

# 3. Reporting Limits

Table 9 lists target analytes and Reported Detection Limit information.

#### 4. Interferences

- **4.1** Contamination may occur in the sampling system if canisters are not properly cleaned before use. Additionally, all other sampling equipment (e.g., pump and flow controllers) must be thoroughly cleaned to ensure that the filling apparatus will not contaminate samples.
- **4.2** System carryover can be a potential problem, particularly for the heavier molecular weight hydrocarbons. Carryover can occur after the analysis of standards or high-level samples. Measures that must be taken to remove this contamination can include the analysis of multiple blanks, and the purging of the autosampler with ambient air.
- **4.3** High moisture content, methane levels and/or carbon dioxide levels may interfere with the chromatography and trapping of target analytes. Dilutions may be performed on these samples; however, the reporting limits will then be elevated.

## 5. Health and Safety

The toxicity or carcinogenicity of each reagent and standard used in this method is not fully established; however, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals must be reduced to the lowest possible level by whatever means available. A reference file of material safety data sheets is available to all personnel involved in the chemical analysis. Additional references to laboratory safety are available in the Chemical Hygiene Plan.

All personnel handling environmental samples known to contain or to have been in contact with municipal waste must follow safety practices for handling known disease causative agents.

All employees performing laboratory procedures must have read and understood the Alpha Analytical Chemical Hygiene Plan. All laboratory procedures must be performed in accordance with the provisions and policies of the manual. All accidents, injuries, spills, or unsafe conditions must be reported immediately to the laboratory manager, and such occurrences must be thoroughly documented.

The analyst must wear a lab coat, gloves, and safety glasses while preparing solutions or handling samples.

Preparation of liquid standards must be performed under a properly functioning fume hood. Preparation and venting of gaseous standards must also be performed under a properly functioning fume hood.

# 6. Sample Collection, Preservation, Shipping and Handling

## 6.1 Sample Collection

- **6.1.1** FSL canister samples can be collected as grab samples or as time-integrated samples. Time-integrated samples can be collected for a maximum of 12 hours using 2.7-liter canisters, or a maximum of 24 hours to 7 days using 6-liter canisters. One liter canisters are typically used for soil vapor sampling with a sampling flowrate of 100-200 ml/min.
  - **6.1.1.1** Grab samples are collected by opening the canister valve and allowing the canister to fill to ambient pressure. This process takes approximately one minute.

- controller. The flow controller, if provided by Alpha, is calibrated prior to sample collection and is documented in the Regulator Tracking Records Logbook (Refer to Alpha SOP A-007 for Canister and Flow Controller Preparation).
- **6.1.2** Tedlar® bag samples typically can be collected as grab or composite samples and may require a pumping system or evacuated box.
- **6.1.3** Upon receipt at the laboratory, all samples are assigned unique laboratory identification numbers, checked for possible discrepancies, etc. (See SOP G-005.)

#### 6.2 Sample Preservation

Canisters-None. Tedlar® bags-should be protected from light.

#### 6.3 Sample Shipping

All samples must be accompanied by a chain of custody form, which documents the date, and time of sample collection.

#### 6.4 Sample Handling

The pressure of all FSL canister samples is measured upon receipt at the laboratory and documented on the FSL Canister Records Logbook (See Alpha SOP A-007). A pressure gauge is attached to the canister inlet, the canister valve is briefly opened and the pressure is recorded. The gauge apparatus used to measure ambient air samples must be separate from that used to measure soil vapor or other matrices known to have elevated levels of VOCs to avoid cross-contamination.

Samples with pressures greater than -15 inches Hg are considered acceptable for analysis.

Samples with less than -15 inches Hg need to be pressurized to > -15 inches Hg in order for the concentrator system to accurately draw the correct volume, resulting in a dilution of the sample. For ambient air samples, the client must be notified prior to sample analysis since this dilution may cause reporting limits to be elevated above project action levels.

Any samples that undergo pressurization prior to analysis are documented in the instrument software. Refer to Section 10.3.3.6 for the calculation of dilution factors due to pressurization of samples.

Refer to SOP G-005 for Sample Management information.

FSL canister and Tedlar® bag samples are stored in the Volatiles Laboratory until analysis has been completed. Tedlar® bag samples are stored in opaque containers.

The recommended holding time for the analysis of FSL canister samples for and TO-15 is 30 days. The recommended holding time for the analysis of Tedlar® bag samples for TO-15 is 48 hours. Tedlar® bag samples requiring TO-15 analysis may be transferred into canisters upon receipt at the laboratory in order to extend the holding time of the sample to 30 days.

# 7. Equipment and Supplies

- 7.1 Microliter syringes: 10, 25, and 500 µL
- 7.2 Gas tight syringes: 1 mL, 5 mL, 25mL, and 50 mL
- 7.3 FSL canisters: 1.0, 2.7, 6.0 and 15 liter
- **7.4 Tedlar® bags:** Various sizes. Alpha supplies 5-Liter sizes. All bags must have polypropylene fittings which are recommended for the analysis of Sulfides and Mercaptans.

#### 7.5 Stop watch

#### 7.6 Sample Concentrator

- **7.6.1** The concentrator system consists of two separate pieces of equipment: (1) Entech Model 7016CA VOC Autosampler, and (2) Entech Model 7100A Cryogenic Concentrator using liquid nitrogen.
- **7.6.2** A vacuum pump (Thomas Industries Model 6073A32) delivers the sample from the autosampler to the cryogenic concentrator FSL-lined steel tubing.

#### 7.7 Gas Chromatograph System

- 7.7.1 Gas chromatograph Hewlett Packard Model 6890N GC
- 7.7.2 Chromatographic column: Restek RTX-1; 60 meters, 0.25 mm ID, 1 micron film thickness
- **7.7.3** Transfer line from column to GC injection port: Hydroguard <sup>™</sup> 0.32 mm capillary tubing connected to column with Restek Vu-Union connector.

#### 7.8 Mass Spectrometer System

- 7.8.1 Mass spectrometer Hewlett Packard Model 5973 or 5975.
- **7.8.2** The mass spectrometer must be capable of scanning from 29 to 270 amu every 3 seconds or less, utilizing 70 volts (nominal) electron energy in the electron impact ionization mode and producing a mass spectrum that meets all the criteria in Table 5 when 50 ng of 4- bromofluorobenzene is injected. For SIM (selective ion monitoring) analysis, the system must be capable of simultaneous SIM/full scan acquisition.
- **7.8.3** Data System EnviroQuant ChemStation G1701 DA Version D.02.00 SPI or later for data acquisition and data processing.

## 8. Reagents and Standards

#### 8.1 DI Water or Carbon-filtered tap water

**8.2 High purity purge and trap grade methanol** (Fisher part # A453-1 or equivalent) for MS source cleaning

## 8.3 Ultra high purity (UHP) helium for the GC/MS system

- 8.4 Ultra high purity (UHP) nitrogen for standard preparation
- **8.5 NIST certified TO-15 gas standards**, purchased from Spectra Gases. Standards are stored at room temperature and expire one year from production date, unless re-certified.
- 8.6 Neat chemicals: Listed in Table 1 and Table 3B, > 98% purity.
- 8.7 Liquid nitrogen: For the concentrator system and/or GC cooling
- **8.8 Zero air (<0.1 ppm total hydrocarbons):** For the concentrator system and standard preparation

#### 8.9 Primary Standards

- **8.9.1** Primary standard mixtures of TO-15 analytes are purchased certified gaseous standards already prepared as well as gaseous standards prepared in the laboratory by injecting neat chemicals into Tedlar® bags. (See Table 1)
- **8.9.2** Table 1 indicates volumes of neat chemicals that are injected into 20 L of zero air or UHP nitrogen to obtain primary standard concentrations for all analytes.
- **8.9.3** Purchased primary standards are assigned a CSS # (commercially supplied standard) upon receipt for tracking purposes. Preparation of primary standards must be entered into the primary standard preparation logbook (Form No.: 117-11).
- 8.9.4 Standards are valid per the manufacturer's expiration date as noted.

#### 8.10 Secondary Standards

- **8.10.1** Prepare secondary standards in canisters using the Entech 4600A Dynamic Diluter at a minimum of two concentration levels. Table 3A and 3B outlines the preparation steps for each secondary standard.
- 8.10.2 Prior to preparation of the standards, verify that an appropriate vacuum exists in the canister (>0.5 psia). Figure 3 demonstrates the standard preparation system. Primary standards prepared in Tedlar bags are injected into a canister (typically 15 L) using an injection tee with a septum or transferred from purchased cylinders via the dynamic diluter.
- **8.10.3** Attach the canister to the ¼ in. fitting at the front of the dynamic diluter, but do not tighten the connection. Attach the transfer lines from the primary standards to the back of the dynamic diluter.

- 8.10.4 Prior to the injection of the gaseous standards, allow the dynamic diluter to equilibrate for a minimum of 60 minutes by allowing the diluent gas to flow at a rate of 200 mL/min and the primary standard to flow a the rate specified in Table 3A for the appropriate standard being prepared (high or low). The flow rate settings are adjusted via the 4600A software, the diluent gas flows through MFC 1 and the calibration standard flows through MFC 2, 3 and 4. Be sure that the isolation valve is open while equilibrating and the vent line is attached to the outlet.
- **8.10.5** After equilibrating the system, close the isolation valve and tighten the fitting on the canister. Set stopwatch or timer to the duration specified in Table 3 for the purchased cylinder primary standards. Open the isolation valve and the canister valve and start the timer.

Equation 1: Flow rate calculation:

$$T_f = V_{std} / F_{std}$$

Where:

 $\begin{array}{l} T_{f} = standard \ transfer \ time, \ minutes \\ V_{std} = standard \ volume, \ mL \\ F_{std} = standard \ flowrate, \ mL/min \end{array}$ 

- **8.10.6** Inject the appropriate amount of Tedlar bag primary standard and the low vapor pressure compounds listed in Table 3B into the injection port tee. This injection must be done while the canister is below atmospheric pressure.
- **8.10.7** When all the primary standards have been added to the canister, pressurize the canister to 30 psia with humidified zero air or nitrogen using the dynamic diluter. This is equivalent to 30 liters of calibration standard in the canister when a 15 L canister is utilized for standard preparation.
  - **NOTE:** Standard canisters prepared for analysis using the autosampler must have a maximum pressure of 30psia to ensure proper and consistent sampling by the instrument.
- **8.10.8** Label the canister accordingly and record the standard preparation in the secondary standard (SS) preparation logbook (Form No.: 117-12).
- 8.10.9 Standards are valid for 3 months.

## 8.11 Internal Standard/BFB Tuning Standard

The internal standard and the bromofluorobenzene (BFB) tuning standard are purchased as a single gas standard at 100 ppbV. The internal standards and BFB are loaded onto the sample trap prior to the sample via a mass flow controller and a mass flow meter. The concentration of the internal standard added is based upon the nominal concentration of sample that is analyzed. If the nominal volume of sample is 250 mL, then 100 mL of the 25 ppbV internal standard mix will yield a true value of 10 ppbV for the internal standards and

BFB. Using equation 7, the ug/m<sup>3</sup> equivalent of BFB injected 716 ug/m<sup>3</sup> or 716 ng/L (MW of BFB = 175). Thus, the total ng injected is:

Total ng = 716 ng/L x 0.025 L = 18 ng

#### 8.12 Instrument Calibration Standards

Calibration standards are prepared by injecting different volumes of the secondary standards into the concentrator/GC/MS system. The low standard will be used to establish the reporting limit for sample analyses. These are described in more detail in Section 10.

## 9. Quality Control

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method.

At a minimum, for each day of analysis, a Continuing Calibration standard, Laboratory Method Blank, Laboratory Control Spike and Laboratory Duplicate must be analyzed. Laboratory Control Spike Duplicate (LCSD) will be analyzed only upon client request.

#### 9.1 Blank(s)

A FSL canister pressurized with nitrogen or zero air and humidified is utilized as the Laboratory Method Blank. The method blank must be free of target analyte contamination at or above the reporting limit. If it is not, the system must be evaluated for possible sources of contamination. Once the source is determined and eliminated, the Blank must be reanalyzed.

A Laboratory Method Blank must be run after samples suspected of being highly contaminated to determine if sample carryover has occurred. If samples have been analyzed using an autosampler, data must be evaluated for potential carryover and reanalyses conducted, as appropriate.

# 9.2 Laboratory Control Sample (LCS) / Laboratory Control Spike Duplicate (LCSD)

**NOTE:** A Laboratory Control Spike Duplicate is only performed when specified by the project requirements and/or upon Client request.

Laboratory Control Spike - A Laboratory Control Spike (LCS) is prepared by spiking an evacuated FSL canister with a different primary standard solution than that used for the calibration or a purchased gaseous standard with the components of interest may be used. The spike recovery must be between 70 and 130%. Up to 10% of the total number of compounds (6 for a standard TO-15 analysis) may exceed this criteria, however no analyte can have a recovery greater than 150% or less than 50%. If the recovery is not within acceptance criteria, the LCS may be analyzed a second time. If the LCS failure continues, the instrument must be recalibrated. Refer to Section 12 for appropriate corrective actions to be taken.

## 9.3 Initial Calibration Verification (ICV)

A mid-range calibration standard must be analyzed after the initial calibration and prior to sample analysis, and must be a different source than that used for the initial calibration. The %Ds must be  $\leq$ 30. If more than 10% of the compounds fail to meet this criterion, or if the %D for any one compound is greater than 50, the instrument is recalibrated. Otherwise, sample analysis may proceed.

## 9.4 Continuing Calibration Verification (CCV)

A mid-range calibration standard must be analyzed prior to sample analysis. This standard is of a different source than that used for the LCS/LCSD pair, typically the same source as the initial calibration standards. The %Ds must be ≤30. If more than 10% of the compounds fail to meet this criterion, or if the %D for any one compound is greater than 50, the instrument must be recalibrated. Otherwise, sample analysis may proceed.

#### 9.5 Matrix Spike

Not applicable.

#### 9.6 Laboratory Duplicate

A Laboratory Duplicate is a replicate analysis of a sample. The RPD of duplicate analyses must not exceed 25. Up to 10% of the target analyte detections may exceed acceptance criteria. If more variation occurs, the sample analysis must be repeated. If an analyte is detected in one analysis at >5x the reporting limit and not detected in the duplicate analysis, the analysis must be repeated. If an analyte is detected in one analysis at <5x the reporting limit and not detected in one analysis at <5x the reporting limit and not detected in one analysis at <5x the reporting limit and not detected in the duplicate analysis, the RPD is not calculable (NC) and the analysis does not have to be repeated. If an analyte is not detected in both the original and duplicate analyses, the RPD is NC. Equation 9 is used to calculate the RPD. The sample chosen for duplicate analysis must be rotated among clients and/or sites. If possible, field duplicates should not be chosen for duplicate analysis, nor should outside air samples if indoor air samples are also included in the analytical batch.

Equation 9: RPD Calculation

 $RPD = (C_s - C_i) / [(C_s + C_d)/2]*100$ 

where:

RPD = relative percent difference

 $C_s$  = concentration in original sample analysis

C<sub>d</sub> = concentration in duplicate sample analysis

## 9.7 Method-specific Quality Control Samples

- **9.7.1 BFB Tune -** A successful BFB spectrum must meet the criteria in Table 5 prior to sample analysis. If a successful BFB spectrum is not obtained, the MS must be retuned and the BFB spectrum re-evaluated prior to analyzing samples.
- **9.7.2** Internal Standards The internal standard area counts of each sample, blank, and Laboratory Control Sample are evaluated against the corresponding continuing calibration standard. The internal standard area counts must be within 60-140% of the continuing calibration standard area counts. The retention times of the internal standards must be within +/- 0.30 min. If the internal standards fall outside this range, the sample, blank, or Laboratory Control Sample must be reanalyzed. In addition, area counts for internal standards for continuing calibration must be within 60-140% recovery of initial calibration.
- **9.7.3 TIC Internal Standards** Internal standards used for the quantitation of TICs must be evaluated by comparing the total ion area counts of the internal standards in the samples to the total ion area counts of the internal standards in

the blanks. The internal standard area counts must be within 50-200% of the blank area counts. If the internal standards fall outside this range, a different internal standard or an estimated internal standard total ion area must be used to quantitate the TIC. This estimate can be done by using the total ion area from a blank or a clean sample within the analytical batch.

## 9.8 Method Sequence

- BFB Tune Check
- Calibration Standards (initial) or Continuing Calibration
- Laboratory Control Sample (may be used as the ICV or CC)
- Laboratory Control Sample Duplicate (if needed)
- Laboratory Method Blank
- Samples
- Laboratory Duplicate

Injections may be made until 24 hours after the injection used to check the BFB tune.

All analytical sequences must be recorded in the instrument software and documented in the instrument logbook.

## 10. Procedure

## 10.1 Equipment Set-up

#### 10.1.1 Canister Cleaning and Certification

Refer to Alpha SOP A-007 for canister and flow controller preparation.

#### 10.1.2 Sample Preparation and Concentration

Ensure the integrity of the canister sample as described in Section 6.5.

Connect the canisters or Tedlar® bag(s) to the Entech 7016CA Autosampler. For FSL canisters: Align the tubing from one of the 16 positions to the canister inlet position. Push the inlet line into the orifice of the canister and hold in place while tightening the fitting finger tight. Turn the stainless steel nut ¼ turn more with a wrench. The canister valves must be closed at this point. For Tedlar® bags: Connect the valve of the Tedlar® bag with the autosampler line using Teflon tubing.

For canister samples, leak check all inlet connections using the leak check procedure included with the Entech software. A report will be generated indicating the change in vacuum over a period of 30 sec. The vacuum must not increase more than 2 psia. Analysis cannot begin until the leak check has passed for each canister being tested and/or the source of the leak has been determined. A copy of this report must be available on file.

Open the canister or bag valves.

Set up the sequence of the Entech system to withdraw 250 mL from each sample. If high concentrations are expected, lower volumes can be used with a minimum of 25 mL using the Entech method alphaTO15.MPT. If volumes lower

than 25 mL are needed, the Entech method TO15\_25X.MPT, which utilizes a slower sampling flow rate can be used, however, this is below the recommended minimum of the instrument and could cause a bias in results.

Recommended concentrator operating parameters are provided in Table 6.

General description of procedure: A 3-stage concentration technique called Microscale Purge and Trap is used to analyze VOCs in air. It is analogous to the Purge and Trap used in water analysis, only on a much smaller scale. The air sample is first concentrated to about a 0.5cc volume in a cryogenic trap. The trap is then heated to 10 degrees C and is held there while slowly passing helium or nitrogen through it to transfer these compounds to a secondary trap. The second trap is usually Tenax at -30 °C. Sweeping the VOCs from the first to the second trap with only 40cc of helium results in a transfer of less than 0.5 µl of water (40cc @ 100% RH @ 10 deg. C) which can be easily handled by benchtop mass spectrometers. The 40 cc transfer volume also serves to flush the CO2 through the Tenax trap. After transfer to the second trap, the VOCs are back-flushed while heating to be further focused on an open-tubular focusing trap (cryofocuser) for rapid injection onto the analytical column. Internal standard is added directly to the first stage cryogenic trap prior to the sample by a mass flow controller (MFC). MFC controlled introduction is advantageous over loop injection as it remains consistent with the mechanism used to measure the sample volume

## 10.2 Initial Calibration

#### 10.2.1 Daily GC/MS Performance Check

- **10.2.1.1** The first analysis of the day is typically a system blank. The GC/MS system is checked to confirm that acceptable performance criteria for bromofluorobenzene (BFB), which is in surrogate mixture, are achieved. These criteria must be met prior to analyzing further standards, blanks and samples.
- **10.2.1.2** A maximum injection of 50 ng must successfully meet the BFB spectrum criteria in Table 5.
- **10.2.1.3** If the spectrum of BFB does not meet the above stated criteria, the analysis must be repeated. If the spectrum of BFB still does not meet these criteria, the GC/MS instrument must be retuned.
- **10.2.1.4** The Daily GC/MS Performance Check must be analyzed every 24 hours or less.

#### 10.2.2 Initial Calibration

**10.2.2.1** Analyze a minimum of five different levels by analyzing various volumes of the secondary standards prepared in Table 3 (Table A-3 for sulfide/mercaptan analysis). The lowest standard will be at or below the reporting limit. If the response is not linear at the lowest level for the higher molecular weight compounds, this point must not be included in the calibration curve for these compounds. As a result, the analysis of more than five levels may be required in order to ensure a minimum of five calibration points for each analyte.

Table 4 lists the calibration standard levels and the volumes of the secondary standards needed to achieve these levels.

**10.2.2.2** The true value of each of these calibration points is determined by applying a dilution factor that is based on the volume of sample extracted from the canister for each calibration point. Assuming that a volume of 250 mL will be the maximum volume extracted from the samples; this will be the "1X" volume. A dilution factor can be calculated using Equation 2.

Equation 2: Calculation of Instrument Dilution Factor

DF = V<sub>1X</sub> / V<sub>actual</sub>

where:

DF = dilution factor

V<sub>1X</sub> = maximum volume sampled, mL

V<sub>actual</sub> = actual volume sampled for samples and standards

- **10.2.2.3** Analyze each calibration standard according to the procedures specified in Section 10. The true value of each calibration point is determined by dividing the concentration of the canister by the dilution factor determined using Equation 2.
- **10.2.2.4** Tabulate the area response of the characteristic ions against the amount for each analyte and internal standard and calculate relative response factors (RRF) for each compound using Equation 3. Perform this calculation for each calibration standard.

Equation 3: Relative Response Factor for Individual Target Analytes

 $RRF = [(A_{EC}) * (C_i)] / [(A_{EI}) * (C_c)]$ 

where:

RRF = relative response factor

 $A_{EC}$  = area count of the extracted ion for the analyte of interest

C, = amount of internal standard (ng)

 $A_{EI}$  = area count of the extracted ion for the associated internal standard  $C_c$  = amount of analyte of interest (ppbV)

Table 7 lists all TO-15 analytes, internal standards and the associated quantitation ion.

Table 8 lists the internal standards and the associated TO-15 analytes.

**10.2.2.5** Calculate the average response factor for each of the target analytes by the following equation  $(AVG_x = SUM(RFs) / \text{total } \# \text{ of } RFs)$ .

Alpha Analytical, Inc. Technical Standard Operating Air Analysis: TO-15	Issue No.:5 R	Rev
Effective Date: May 22, 2009 10.2.2.6	Issue Date:April 22, 2 Calculate the percent relative standard deviation (%RSD) of the response factors over the secondary range of the curve for each of the ta analytes using Equation 4.	ons
	Equation 4: Percent Relative Standard Deviation	
	%RSD = [(SD <sub>n</sub> - 1) / (AVG <sub>x</sub> )] * 100]	
	where:	
	%RSD = percent relative standard deviation SD <sub>n</sub> -1 = standard deviation (n-1 degrees of freedom) AVG <sub>x</sub> = average response factor from the initial calibration curve	e
	This task can also be accomplished using the quantitation soft provided by the instrument manufacturer.	war
10.2.2.7	If the %RSD is <30 for each analyte, linearity can be assumed for associated target analyte and sample analysis may proceed.	r th
	If the %RSD is >30 for any analyte, the integrations must be evaluated the calculations verified. If a %RSD <30 cannot be achieved, acceptable for 10% of the total analytes to be above 30%, but below RSD. Before acceptance of such a Calibration Curve, it must be confin with the approval of the Section Supervisor and/or the Project Man that these analytes are typically and historically "trouble" analytes or " performers" (typically compounds listed in Table 3B), and that all C and Project Data Quality Objectives (DQOs) will still be met w analyzing samples using this calibration.	it 50 <sup>4</sup> rme age 'poe Clie
	However, if either >10% of the target analytes are >30% and < 50% OR if any analyte is >50% RSD (with the exception of Table 3B analy the instrument must be recalibrated prior to processing any samples.	
	Calibration points may be removed from the calibration curve to mee 30% RSD criteria, so long as five consecutive points remain in calibration curve, and the following procedure is followed:	
	Remove high level calibration points	
	• Remove low-level calibration points; reporting limits will need to elevated, however.	o t
	• If calibration points in the mid-level range need to be removed entire calibration level must be removed from the calibration curve.	
10.2.2.8	If the %RSD >30, a calibration curve is generated using the Envirod quantitation software. The correlation coefficient (linear) for the calibr curve must be greater than 0.995. If these criteria cannot be met, pre a new set of calibration standards and recalibrate the instrument. No Quadratic calibration in any form is <u>not</u> acceptable.	atic pa
	Authorization from the department supervisor is required prior to u linear regression calibration. Linear regression is only allowed if ce criteria listed below are met:	

	points.
	<ul> <li>The curve must be plotted and printed and turned in with the raw data.</li> </ul>
	<ul> <li>A calibration standard must be analyzed at the low point of the curve. Recovery of the low point standard must be 60-140% using the linear regression curve.</li> </ul>
	<ul> <li>The recovery of the compound for the continuing calibration / LCS must be within 70-130%.</li> </ul>
10.2	2.2.9 The reference spectra for all target analytes are reviewed for both assignments and purity for all instruments. In addition, this process of reviewing all spectra continues whenever a new calibration is completed.
10.2	2.2.10 Internal Standard Criteria for Initial Calibration Curve
	The mean response for each internal standard compound is calculated over the initial calibration range. The area response at each calibration level must be within 40% of the mean area response over the initial calibration range for each internal standard. This criteria must be met prior to sample analysis.
	All of these criteria must be met prior to sample analysis.
10.3 Equip	ment Operation and Sample Processing
10.3.1	GC Conditions

Oven program:25° C, hold for 5.0 minutes, then:Ramp 1: 100° C at 8.0° C / min.; hold for 0.0 minRamp 2: 220° C at 25° C / min.; hold for 4.0 minGas FlowsHelium carrier gas flow program: 2.0 mL/min for entire run (23.18 min)

#### Sample Injection

Injection mode:	split
Injection port temperature:	250 <sup>0</sup> C
Inlet pressure:	27.3 psi
Total flow:	39.3 mL/min
Split ratio:	17.3
Split flow:	34.6 mL/min
Gas saver flow:	OFF

#### 10.3.2 MS Conditions

Temperature of MSD transfer line: 240<sup>°</sup> C Temperature of MS Quad: 150<sup>°</sup> C Temperature of MS Source: 230<sup>°</sup> C Solvent Delay: 3.0 minutes Scanning Parameters: 29-270 amu until 10 min, scan rate = 5.52 scans/sec; then 35-270 amu, scan rate = 3.1 scans/sec. Threshold = 150. Sampling rate = 2. EM offset-variable to achieve response of 200K area counts (+/- 25K) for the internal standard bromochloromethane.

#### 10.3.3 GC/MS ANALYSIS

- 10.3.3.1 The Entech 7100A Concentrator is programmed to the specific analytical conditions listed in Table 6 (Entech method alpha\_TO15.MPT) and the GC/MS parameters are set to those listed in Sections 10.3.1 and 10.3.2. (Enviroquant method TO15-SFS.M (SIM and full scan) or TO15.M (full scan only)).
- **10.3.3.2** An system blank is performed. The BFB spectrum is evaluated.
- **10.3.3.3** A continuing calibration and/or a laboratory control spike is analyzed. See sect. 9.2 for acceptable criteria
- 10.3.3.4 A Laboratory Method Blank is analyzed. The Laboratory Method Blank consists of the analysis of 250 mL from a canister of humidified nitrogen or zero air. The method blank must be free of target analyte contamination at or above the reporting limit.
- **10.3.3.5** A 250-mL aliquot of sample is preconcentrated on the Entech 7100A concentrator and injected onto the GC column.
- 10.3.3.6 Instrument Dilutions and Sub-Atmospheric Sample dilutions
  - 10.3.3.6.1 For dilutions, smaller sample volumes (<250 mL) are analyzed. The smallest volume that can be analyzed with accuracy is 25 mL. The dilution factor is accounted for by entering the volume analyzed in the sample calculation discussed in Section 10.2.2.2 (Equation 2). A 10 mL aliquot may be analyzed, if the sample is withdrawn from the canister at a lower flowrate using the Entech method TO15\_25X.MPT.
  - **10.3.3.6.2** Samples that arrive at the laboratory with pressures below -15 inches Hg must be pressurized with zero air to greater than -15 inches Hg, as discussed in Section 6.4. This pressurization results in a dilution factor. The dilution factor is calculated using Equation 6, and the canister dilution spreadsheet (Form No.: 117-05). Attach a green tag to the canister with the pressurization information (initial pressure and final pressure) recorded on the tag.

**Equation 6**: Dilution Factor for Pressurization of Subatmospheric Samples: Three Steps

Step 1: Calculate the volume in the canister prior to pressurization (Assume a 2.7-liter canister is used).

 $V_{ci} = 2.7 * P_1 / 14.696$ 

Step	2:	Calculate	the	volume	in	the	canister	after	
press	uriz	ation.							

 $V_{cf}$  = 2.7 \*  $P_F$  / 14.696

Step 3: Calculate the dilution factor.

 $DF = V_{cf} / V_{ci}$ 

where:

V<sub>ci</sub>= volume of air in canister prior to pressurization, L

P<sub>I</sub> = pressure reading of canister prior to pressurization (psia)

V<sub>cf</sub> = volume of air in canister after pressurization, L

 $P_F$  = pressure reading of canister after pressurization (psia)

DF = dilution factor

14.696 = atmospheric pressure (psia)

- **10.3.3.7** If samples require larger dilutions than pressurization and instrument dilutions, a syringe dilution into a Tedlar bag with a known volume of nitrogen or additional canister is required.
- 10.3.3.8 Fit a VCO adapter with a septa to the pressurized sample canister. With a gastight syringe remove appropriate sample size for dilution. Allow sample to flow through syringe for 1 2 seconds to flush syringe prior to volumizing. Inject the sample aliquot into a Tedlar bag. If using an evacuated canister, connect the canister to an injection port tee (see Figure 3) attached to the dynamic diluter. Inject the aliquot of sample while a steady stream of Nitrogen is flowing into the dilution canister. Pressurize this canister to 30psia. Attach a green tag to the canister with dilution information recorded on the tag. Use the dilution calculation worksheet (Form No.: 117-05) to calculate resulting dilutions.

#### 10.3.4 Qualitative Identifications

- **10.3.4.1** An analyst competent in the interpretation of mass spectra must identify the target analytes by comparison of the sample mass spectrum to the mass spectrum of the standard. Two criteria must be satisfied to verify the identification: (1) elution of the component in the sample at the same GC relative retention time (RRT) as the component in the standard, and (2) agreement of the sample component and standard component mass spectra.
- **10.3.4.2** For establishing correspondence of the GC RRT, the RRT of the component in the sample must compare within ±0.06 RRT units of the RRT of the component in the standard. If co-elution of interfering components prohibits accurate assignment of the sample component RRT from the total ion chromatogram, the RRT must be assigned using extracted ion current profiles for the ion unique to the component of interest.
- 10.3.4.3 For comparison of the standard and sample component mass spectra, mass spectra of standards obtained on the GC/MS under the same

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	instrument conditions are required. Once may be used for identification and reference	•
10.3.4.4	The requirements for qualitative verification are as follows:	n by comparison of mass spectra
	<ul> <li>All ions present in the standard mas greater than 10% (most abundant ion must be present in the sample spectru</li> </ul>	n in the spectrum equals 100%)
	The relative intensities of ions spe between the standard and sample spe	그는 것은 것은 것은 것을 하는 것을 하는 것을 가지 않는 것을 하는 것을 수가 있다.
	<ul> <li>lons greater than 10% in the sample s accounted for by the analyst making the</li> </ul>	
	Table 7 lists the primary and secondary ion	ns for all analytes.
10.3.4.5	Tentatively identified compounds (TICs A library search may be performed for no the purpose of tentative identification, as spectra are compared to the Nationa Technology Mass Spectral Library, and a by the analyst. Computer generated libra normalization routines that would misre spectra when compared to each other.	on-target sample components for s requested by the client. Mass al Institute of Standards and qualitative match is determined ry search routines must not use
10.3.4.6	Guidelines for making tentative identification	on:
	<ul> <li>Relative intensities of major ions in greater than 20% of the most abund sample spectrum.</li> </ul>	
	• The relative intensities of the major ion	is must agree within ±30%.
	<ul> <li>Molecular ions present in the referen- the sample spectrum.</li> </ul>	ce spectrum must be present in
	<ul> <li>lons present in the sample spectrum to must be reviewed for possible backgr of coeluting compounds.</li> </ul>	-
	<ul> <li>If, in the technical judgment of the specialist, no valid tentative identificate will be reported as "Unknown". The specialist should give additional compound, if possible (i.e., unknown unknown acid, unknown chlorinated c weights can be distinguished, include the</li> </ul>	ion can be made, the compound ne mass spectral interpretation classification of the unknown aromatic, unknown hydrocarbon, ompound). If probable molecular

#### 10.4 Continuing Calibration

#### 10.4.1 Calibration Verification

- **10.4.1.1** The initial calibration must be verified through the analysis of an Initial Calibration Verification (ICV) sample. (The ICV may also be used to satisfy LCS requirements.) This analysis must be performed every time an initial calibration is performed.
- **10.4.1.2** The ICV must be prepared using a purchased gaseous standard (from a separate vendor if available) with the components of interest in an evacuated FSL canister. Follow the standard preparation procedure for the calibration standards outlined in Section 8.0. The standard must be prepared at or below the midpoint of the calibration curve.

See section 9.3 for acceptable criteria.

#### 10.4.2 Continuing Calibration

- **10.4.2.1** A continuing calibration check must be performed daily prior to sample analysis. The continuing calibration standard must be one of the initial calibration levels.
- **10.4.2.2** Analyze a calibration standard that is at the midpoint of the calibration curve.
- **10.4.2.3** The LCS standard may be utilized as the continuing calibration check, provided that all target analytes of interest are present in the LCS standard.
- **10.4.2.4** Calculate the percent difference (%D) of the continuing calibration response factor from the initial calibration average response factor using Equation 5.

Equation 5: Percent Difference

% D =  $[(C_{found})-(C_{true}) / (C_{true})] * 100$ 

where:

%D = percent difference

 $C_{found}$  = amount of the analyte detected in the standard (ppbV)  $C_{true}$  = true amount of the analyte in the standard (ppbV)

This task can also be accomplished using the quantitation software provided by the instrument manufacturer.

**10.4.2.5** The %Ds must be <30. If more than 10% of the compounds fail to meet this criterion, or if the %D for any one compound is greater than 50, the instrument must be recalibrated.

#### **10.5 Preventive Maintenance**

send out the MFC for re-certification annually

Ion source cleaning

Rough pump oil changed annually

# 11. Data Evaluation, Calculations and Reporting

## **11.1 Calculations**

11.1.1 Individual Target Analytes: The average response factor from the initial calibration is used to calculate the amount of analyte detected in the sample analyses. Standards are prepared on a ppbV basis, so if no dilution is performed, values can be reported from the quantitation report without any calculations. Dilution factors are calculated using Equation 2. Equation 7 shows the conversion of ppbV to µg/m<sup>3</sup>.

**Equation 7**: Conversion of ppbV to µg/m<sup>3</sup>

 $\mu$ g/m<sup>3</sup> = (ppbV) \* MW / 24.47

where:

24.47 = molar gas constant (g/g-mole)

MW = molecular weight of the compound of interest (Table 1 and 2 lists the molecular weights of the target analytes)

**11.1.2 TICS:** An estimated amount for the TIC is calculated using the total area of the TIC and the total area of the nearest internal standard using a response factor of 1.000 (Equation 8). The calculation must be performed with the closest eluting internal standard which does not exhibit interference from other analytes.

Equation 8: Calculation of TIC Results in ug/m<sup>3</sup>

 $ug/m^3 = [(A_T) * (C_{IS})] / [(A_{IS-T}) * (1.000)]$ 

where:

 $A_T$  = total ion area of the TIC to be measured

C<sub>IS</sub> = amount of the internal standard

A<sub>IS-T</sub> = total ion area of the closest eluting internal standard

The integration of target analytes and internal standards must be performed from valley to valley.

#### 11.2 Data Package

#### 11.2.1 Canister Cleaning Information

A copy of the data for the batch certification analysis associated with the FSL canisters must be on file. The raw data must include a sample chromatogram, quantitation report, and spectra of all positive results.

#### 11.2.2 BFB Tune Checks

Tune checks must be included for all days of analysis, including initial calibration. Raw data must include the chromatogram, mass spectra, and summary of relative abundances of the BFB ions.

#### 11.2.3 Calibration Data

- Initial calibration summary (including average response factors, %RSDs, and copies of calibration curves, if appropriate) for target analytes and all calibration chromatograms must be on file.
- Continuing calibration summaries (including %Ds) for individual analytes.
- Chromatograms and quantitation reports associated with all standards used, in the initial and continuing calibrations.

#### 11.2.4 QA/QC

- Summary of internal standard responses and % recoveries vs. the continuing calibration.
- Quantitation report and chromatogram for laboratory control spike (and laboratory control spike duplicate, if requested).
- Quantitation reports, chromatograms, and spectra of positive results for all blanks.
- Copy of the instrument runlog.

#### 11.2.5 Sample Data

- Quantitation reports, chromatograms, and spectra of positive results for all samples, and duplicates.
- A copy of the canister dilution worksheet, Form No.: 117-05 (if any canister pressurizations or dilutions are performed).

# 12. Contingencies for Handling Out-of-Control Data or Unacceptable Data

The laboratory must maintain records to document the quality of data that is generated. Ongoing data quality checks are compared with established performance criteria to determine if the results of analyses meet the performance characteristics of the method. When results indicate atypical method performance, a calibration verification standard is used to confirm the measurements were performed in an in-control mode of operation.

Holding time exceedence and/or container damage is noted on the Sample Delivery Group form.

Perform routine preventative maintenance following manufacturer's specification. Record all maintenance in the instrument logbook.

Review of standards, blanks and standard response for acceptable performance occurs for each batch of samples. Record any trends or unusual performance on a nonconformance action form.

If the CV, LCS or LCSD recovery of any parameter falls outside the designated acceptance range, the laboratory performance for that parameter is judged to be out of control, and the problem must be immediately identified and corrected. The analytical result for that parameter in the unspiked samples is suspect and is only reported for regulatory compliance purposes with the appropriate Narratives. Immediate corrective action includes reanalyzing all affected samples by using any retained sample before the expiration of the holding time.

## **13. Method Performance**

# 13.1 Method Detection Limit Study (MDL) / Limit of Detection Study (LOD) / Limit of Quantitation (LOQ)

The laboratory follows the procedure to determine the MDL, LOD, and/or LOQ as outlined in Alpha SOP/08-05. These studies performed by the laboratory are maintained on file for review.

#### **13.2 Demonstration of Capability Studies**

Refer to Alpha SOP/08-12 for further information regarding IDC/DOC Generation.

#### 13.2.1 Initial (IDC)

The analyst must make an initial, one-time, demonstration of the ability to generate acceptable accuracy and precision with this method, prior to the processing of any samples.

#### 13.2.2 Continuing (DOC)

The analyst must make a continuing, annual, demonstration of the ability to generate acceptable accuracy and precision with this method.

## 14. Pollution Prevention and Waste Management

Refer to Alpha's Chemical Hygiene Plan and Waste Management and Disposal SOP for further pollution prevention and waste management information.

## **15. Referenced Documents**

Chemical Hygiene Plan

SOP/08-05 MDL/LOD/LOQ Generation

SOP/08-12 IDC/DOC Generation

SOP G-006 Hazardous Waste & Sample Disposal

Form 117-05: Canister Dilution Worksheet Template

Form 117-11: Primary Standard Preparation Log

Form 117-12: Secondary Standard Preparation Log

#### 16. Attachments

Table 1	TO-15 Tedlar® Bag Stock Standard Preparation
Table 2A	TO-15 Purchased Stock Standard Cylinders
Table 2B	TO-15 Custom Mix Purchased Stock Standard Cylinder
Table 3A	Summary of Working Standard Preparation
Table 3B	Preparation of Calibration Standards for Low Vapor Pressure Compounds
Table 4	Calibration Standard Levels
Table 5	BFB Key lons and Abundance Criteria
Table 6	Entech7100A/7016CA Operating Parameters
Table 7	Quantitation and Secondary lons for TO-15 Analytes and Internal Standards
Table 8	Internal Standards and the Associated Target Analytes
Table 9A	TO-15 Target Analytes and Reporting Limits-Standard List
Table 9B	TO-15 Target Analytes and Reporting Limits-Additional Analytes
Figure 3	FSLCanister Standard Preparation System
Appendix A	Cold Trap Dehydration technique (CTD) for Analysis of Sulifides and Mercaptans
Appendix B	Data Acquisition Parameters and Analysis Modifications for Conducting SIM

#### DEFINITIONS

Analysis

**Absolute canister pressure -** Pg + Pa, where Pg = gauge pressure in the canister (psig) and Pa = barometric pressure.

**Absolute pressure -** Pressure measured with reference to absolute zero pressure (as opposed to atmospheric pressure), usually expressed as kPA, mm Hg, or psia (pounds per square inch absolute).

**Cryogen** - The refrigerant used to obtain very low temperatures in the cryogenic trap of the analytical system. A typical cryogen is liquid nitrogen (bp =  $-196^{\circ}$ C).

**Gauge pressure -** Pressure measured above atmospheric pressure (as opposed to absolute pressure). Zero gauge is equal to ambient atmospheric (barometric) pressure. Units = psig (pounds per square inch gauge).

**ppmV** – parts per million on a volume basis.

**ppbV** – parts per billion on a volume basis

psia – pounds per square inch absolute

**Relative retention time (RRT)**– retention time (RT) ratio of the target analyte and the internal standard used to quantitate (RT target / RT internal standard).

## Table 1

				Maria Rever
COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected*	FINAL ppmV
Indan	118.18	965	5.0	50.0
Indene	116.16	996	4.8	50.4
1,2,3-Trimethylbenzene	120.19	894	5.5	50.1
Thiophene	84.14	1051	3.3	50.4
2-Ethylthiophene	112.19	990	4.7	50.7
2-Methylthiophene	98.17	1014	4.0	50.6
3-Methylthiophene	98.17	<b>10</b> 16	4.0	50.6
Acetaldehyde	44.05	785	11.5	250.7
2,4,4-Trimethyl-2-pentene	112.21	722	6.4	50.4
2,4,4-Trimethyl-1-pentene (diisobutylene)	86.5	708	6.5	50.2
Halothane **	197.38	1872	4.3	49.9

#### **TO-15 Tedlar® Bag Stock Standard Preparation**

\* All neat chemicals are injected into a Tedlar® bag containing 20 Liters of zero air or UHP nitrogen.

\*\* Halothane reported via SIM analysis only.

See Table A-1 for sulfide/mercaptan stock standard preparation

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# Table 2A

1	[O-15]	TO-15 Primary Standard Mix #2						
COMPOUND	MOL WGT	Conc. ppmV	COMPOUND	MOL WGT	Conc. ppmV	COMPOUND	MOL WGT	Conc. ppmV
dichlorodifluoromethane	120.92	1.0	cis-1,3-dichloropropene	110.97	1.0	Propylene	42.08	1.0
chloromethane	50.49	1.0	trans-1,3-dichloropropene	110.97	1.0	1,3-butadiene	54.09	1.0
Freon-114	170.92	1.0	1,1,2-trichloroethane	133.41	1.0	Vinyl bromide	106.96	1.0
vinyl chloride	62.5	1.0	toluene	92.14	1.0	Acetone	58.08	1.0
bromomethane	94.94	1.0	1,2-dibromoethane	187.87	1.0	Isopropyl alcohol	60.1	1.0
chloroethane	64.52	1.0	tetrachloroethene	165.83	1.0	Carbon disulfide	76.14	1.0
trichlorofluoromethane	137.37	1.0	chlorobenzene	112.56	1.0	3-chloropropene	76.53	1.0
1,1-dichloroethene	96.94	1.0	ethylbenzene	106.17	1.0	Trans-1,2- dichloroethene	96.94	1.0
methylene chloride	84.93	1.0	m-xylene	106.17	1.0	Methyl-tert butyl ether	88.15	1.0
Freon-113	187.38	1.0	p-xylene	106.17	1.0	Vinyl acetate	86.09	1.0
trans-1,2-dichloroethene	98.96	1.0	styrene	104.15	1.0	2-butanone (MEK)	72.11	1.0
1,1-dichloroethane	96.94	1.0	1,1,2,2-tetrachloroethane	167.85	1.0	Hexane	86.18	1.0
cis-1,2-dichloroethene	96.94	1.0	o-xylene	106.17	1.0	Ethyl acetate	88.11	1.0
chloroform	119.38	1.0	1,3,5-trimethylbenzene	120.2	1.0	Tetrahydrofuran	72.11	1.0
1,2-dichloroethane	98.96	1.0	1,2,4-trimethylbenzene	120.2	1.0	Cyclohexane	84.16	1.0
1,1,1-trichloroethane	133.41	1.0	1,3-dichlorobenzene	147.0	1.0	Bromodichloromethane	163.83	1.0
benzene	78.11	1.0	1,4-dichlorobenzene	147.0	1.0	1,4-dioxane	88.11	1.0
carbon tetrachloride	153.82	1.0	1,2-dichlorobenzene	147.0	1.0	2,2,4-trimethylpentane	114.23	1.0
1,2-dichloropropane	113	1.0	1,2,4-trichlorobenzene	181.45	1.0	Heptane	100.21	1.0
trichloroethene	92.14	1.0	hexachlorobutadiene	260.76	1.0	4-methyl-2-pentanone (MIBK)	100.16	1.0
						2-hexanone	100.16	1.0

## **TO-15 Purchased Primary Standard Mix**

All mixes currently purchased from Spectra Gases

208.29

252.75

126.59

120.2

1.0

1.0

1.0

1.0

Bromoform Benzyl chloride

4-ethyl toluene

Dibromochloromethane

## Table 2B

## TO-15 Purchased Custom Mix

TO-15 Custom Standard Mix										
COMPOUND	MOL WGT	Conc. ppmV	COMPOUND	MOL WGT	Conc. ppmV					
Propane	44.10	1.0	n-Octane	114.23	1.0					
Chlorodifluoromethane	86.47	1.0	1,1,1,2-Tetrachloroethane	167.85	1.0					
Methanol	32.04	5.0	1,2,3-Trichloropropane	147.43	1.0					
n-Butane	58.12	1.0	Nonane	128.26	1.0					
Dichlorofluoromethane	102.92	1.0	Isopropylbenzene	120.19	1.0					
Ethanol	46.07	5.0	Bromobenzene	157.01	1.0					
Acetonitrile	41.05	1.0	2-Chlorotoluene	126.58	1.0					
Acrolein	56.10	1.0	n-Propylbenzene	120.19	1.0					
n-Pentane	72.20	1.0	4-Chlorotoluene	126.58	1.0					
Acrylonitrile	53.10	1.0	tert-Butylbenzene	134.20	1.0					
Ethyl Ether	74.12	1.0	n-Decane	142.28	1.0					
tert-Butyl Alcohol	74.12	1.0	sec-Butylbenzene	134.20	1.0					
2,2-Dichloropropane	112.99	1.0	p-Isopropyltoluene	134.22	1.0					
Di-Isopropyl Ether	102.17	1.0	n-Butylbenzene	134.20	1.0					
Tert-Butyl Ethyl Ether	102.20	1.0	1,2-Dibromo-3- chloropropane	236.33	1.0					
1,1-Dichloropropene	110.97	1.0	n-Undecane	156.31	1.0					
Tert Amyl Methyl Ether	102.17	1.0	Naphthalene	128.17	1.0					
Dibromomethane	173.83	1.0	n-Dodecane	170.33	1.0					
1,3-Dichloropropane	112.99	1.0	1,2,3-Trichlorobenzene	181.45	1.0					
n-Butyl Acetate	116.16	1.0								

All mixes currently purchased from Spectra Gases

# Table 3A

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume canister (L)	Final Concentration ppbV
Seco	ndary standards	prepared using d	ynamic dilution s	system (Entech 4	600 <b>A</b> )
Tedlar® bag primary standard	50	60 mL	Syringe Injection	30	100
TO-15, & Custom Mix	1.0	3000 mL	50 mL/min for 60 min.	30	100
Tedlar® bag primary standard	50	12 mL	Syringe Injection	30	20
TO-15, & Custom Mix	1.0	600 mL	50 mL/min for 12 min.	30	20
Tedlar® bag primary standard	50	3 mL	Syringe Injection	30	5.0
TO-15, & Custom Mix	1.0	150 mL	50 mL/min for 3 min.	30	5.0
Tedlar® bag primary standard	50	0.6 mL	Syringe Injection	30	1.0
TO-15, & Custom Mix	1.0	30 mL	20 mL/min for 1.5 min.	30	1.0
	Seconda	ary standards pre	pared via serial o	lilution	
100 ppbV ICAL mix	0.1	60 mL	Syringe Injection	30	0.2
100 ppb∨ ICAL mix	0.1	12 mL	Syringe Injection	30	0.04 *

\* This calibration standard is used for TO-15 SIM analysis only (see Appendix B).

## Table 3B

## Preparation of Calibration Standards for Low Vapor Pressure Compounds

COMPOUND (solids)	Vapor Pressure* (P), atm	Molecular Weight	Volume (V) extracted, mL	Gas Constant (R) (L atm/gm mol K)	T, °K	n**	Final Volume, L	mg	ug/m <sup>3</sup>	Vdqq
1-methylnaphthalene	7.70E-05	141.20	0.40	0.082057	298.1	1.259E- 09	30	0.0001777	5.9	1.0
1-methylnaphthalene	7.11E-05	142.20	2.1	0.082057	298.1	6.101E- 09	30	0.0008675	28.9	5.0
1-methylnaphthalene	7.11E-05	142.20	8.4	0.082057	298.1	2.44E- 08	30	0.00347	115.7	20
1-methylnaphthalene	7.11E-05	142.00	42	0.082057	298.1	1.22E- 07	30	0.0173255	577.5	100
2-methylnaphthalene	7.70E-05	141.00	0.40	0.082057	298.1	1.259E- 09	30	0.0001775	5.9	1.0
2-methylnaphthalene	8.96E-05	142.00	1.7	0.082057	298.1	6.228E- 09	30	0.0008843	29.5	5.08
2-methylnaphthalene	8.96E-05	142.00	6.8	0.082057	298.1	2.491E- 08	30	0.0035373	117.9	20.3
2-methylnaphthalene	8.96E-05	142.00	34	0.082057	298.1	1.246E- 07	30	0.0176867	589.6	101.6
1,2,4,5- tetramethylbenzene	4.23E-04	134.22	0.07	0.082057	291.5	1.236E- 09	30	0.000166	5.5	1.0
1,2,4,5- tetramethylbenzene	4.23E-04	134.22	0.35	0.082057	291.5	6.182E- 09	30	0.0008298	27.7	5.0
1,2,4,5- tetramethylbenzene	4.23E-04	134.22	1.4	0.082057	291.5	2.473E- 08	30	0.0033191	110.6	20
1,2,4,5- tetramethylbenzene	4.23E-04	134.22	7.0	0.082057	291.5	1.236E- 07	30	0.0165954	553.2	101
benzothiophene	7.70E-05	133.20	0.38	0.082057	291.5	1.223E- 09	30	0.0001629	5.4	1.0
benzothiophene	7.70E-05	134.20	1.9	0.082057	291.5	6.115E- 09	30	0.0008206	27.4	5.0
benzothiophene	7.70E-05	134.20	7.6	0.082057	291.5	2.446E- 08	30	0.0032824	109.4	20.0
benzothiophene	7.70E-05	134.20	38	0.082057	291.5	1.223E- 07	30	0.016412	547.1	100

Approximately 5.0 g of solid material was allowed to stand in a 250 mL jar w/ septa cap for 30 min prior to removal of vapor phase aliqubt. The aliquot was then spiked directly into secondary standard.

All vapor pressure values from Lange's Handbook of Chemistry & Physics w/ the exception of 1,2,4,5-tetramethylbenzene. The value was derived using Antoine Equation below:

Antoine Equation used to calculate vapor pressure										
	p, mm Hg	log p	А	В	С	T, C	Т, К			
1,2,4,5-tetramethylbenzene	0.32108333	-0.4933822	7.08	1672.43	201.43	19.4	292.55			

## Table 4

C	alibration Stand	lard Levels
Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.20	250 mL of 0.2 ppb∀ sec. standard
2	0.50	125 mL_of 1.0 ppb∀ sec. standard
3	1.0	250 mL_of 1.0 ppb∀ sec. standard
4	2.5	125 mL_of 5.0 ppb∀ sec. standard
5	5.0	250 mL of 5.0 ppbV sec. standard
6	10	125 mL_of 20 ppb∀ sec. standard
7	20	250 mL of 20 ppbV sec. standard
8	50	125 mL_of 100 ppb∀ sec. standard
9	100	250 mL_of 100 ppb∀ sec. standard

# Calibration Standard Levels

## Table 5

## BFB Key lons and Abundance Criteria

Mass	Ion Ábundance Criteria
50	8.0-40.0 percent of the base peak
75	30.0-66.0 percent of the base peak
95	Base peak, 100 percent relative abundance
96	5.0-9.0 percent of the base peak
173	Less than 2.0% of mass 174
174	50.0 to 120.0% of mass 95
175	4.0-9.0 percent of mass 174
176	Greater than 93.0 percent but less than 101.0 percent of mass 174
177	5.0-9.0 percent of mass 176

# Table 6

Module 1 (Glass Bea	d Trap)
Parameter	Setting
Trapping Temperature	-150° C
Internal standard volume	100 mL
Internal standard flow rate	50 mL / min
Sample volume	250 mL
(may vary depending on sample concentrations)	
Sample flow rate, mL / min	150 mL / min
Sample flow rate, mL / min (if volume is < 25 mL)	15 mL / min
Preheat Temperature	20° C
Desorb Temperature	20° C
Bake Temperature	220° C
Bake Time	5 min
Module 1 to Module 2 transfer volume / rate	40 cc @ 10 cc/min
Module 2 (Tenax t	rap)
Parameter	Setting
Trapping Temperature	-10° C
Desorb Temperature	220° C
Bake Temperature	220° C
Module 2 to Module 3 desorb time	3.5 min
Module 3 (Cryofocu	isser)
Parameter	Setting
Cryofocusing Temperature	-160° C
Desorb Temperature	Approx. 70° C
Module 3 to GC desorb time	1.5 min
Bake temperature / event	Approx. 90° C / event 3
Delay time	13 min

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Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)	Compound	Quant. Ion	Sec. Ion(s)
bromochloromethane	49	130	1,4-difluorobenzene	114	63	bromoform	173	171, 175
chlorodifluoromethane	51	67	n-hexane	57	43, 86	styrene	104	103, 78
propylene	41	39, 42	diisopropyl ether	87	45, 59	1,1,2,2-tetrachloroethane	83	85
propane	29	43,39	ethyl acetate	61	43, 70	o-xylene	91	106
dichlorodifluoromethane	85	87	2,2-dichloropropane	77	41, 97	1,2,3-trichloropropane	75	39, 110
chloromethane	50	52	tetrahydrofuran	42	71, 72	nonane	43	57, 128
Freon-114	85	87, 135	tert-butyl ethyl ether	59	87, 57	bromofluorobenzene	95	75, 174
methanol	31	32,29	1,2-dichloroethane-D4	65	67, 102	isopropylbenzene	105	120
vinyl chloride	62	64,	1,1,1-trichloroethane	97	61, 119	bromobenzene	77	156
1,3-butadiene	54	39	1,1-dichloropropene	75	39,110	2-chlorotoluene	126	91
butane	43	41,58	benzene	78	52	n-propylbenzene	120	91
bromomethane	94	96	carbon tetrachloride	117	119, 82	4-chlorotoluene	91	126
chloroethane	64	66	cyclohexane	56	84, 41	4-ethyl toluene	105	91, 120
dichlorofluoromethane	67	69, 47	tert-amyl methyl ether	73	43, 87	1,3,5-trimethylbenzene	105	91, 120
ethanol	31	45	dibromomethane	93	95, 174	tert-butlybenzene	119	134
acetonitrile	41	40	1,2-dichloropropane	63	39, 62	1,2,4-trimethylbenzene	105	91, 120
vinyl bromide	106	108	bromodichloromethane	83	85, 129	decane	57	43, 142
acrolein	56	55,29	trichloroethene	130	132, 97	benzyl chloride	91	126
acetone	43	58	1,4-dioxane	88	58	1,3-dichlorobenzene	146	75, 111
trichlorofluoromethane	101	103	2,2,4-trimethylpentane	57	41, 99	1,4-dichlorobenzene	146	75, 111
isopropyl alcohol	45	59	n-heptane	43	57, 100	sec-butylbenzene	105	134
acrylonitrile	53	52,51	2,4,4-trimethyl-1-pentene	57	41, 112	p-isopropyltoluene	119	134
pentane	43	57,72	cis-1,3-dichloropropene	75	39, 77	1,2-dichlorobenzene	146	75, 111
ethyl ether	31	59,45	4-methyl-2-pentanone	43	58, 100	n-butylbenzene	91	134
1,1-dichloroethene	61	96, 63	2,4,4-trimethyl-2-pentene	55	97, 112	1,2-dibromo-3-chloropropane	75	39, 157
Tertiary butyl Alcohol	59	41, 43	trans-1,3-dichloropropene	75	39, 77	undecane	57	43, 71, 156
methylene chloride	49	84	1,1,2-trichloroethane	97	61, 83	dodecane	57	43
3-chloropropene	41	39, 76	Thiophene	84	45, 58	1,2,4-trichlorobenzene	180	109, 145
carbon disulfide	76	44	chlorobenzene-D5	54	82, 117	naphthalene	128	102
Freon 113	101	85, 151	toluene	91	92	1,2,3-trichlorobenzene	180	109, 182
trans-1,2-dichloroethene	61	96, 98	toluene-D8	98	100	hexachlorobutadiene	225	118, 260
1,1-dichloroethane	63	65	1,3-dichloropropane	76	41,49	2-methylthiophene	97	45, 98
MTBE	73	57, 43	2-hexanone	43	58, 100	3-methylthiophene	97	45, 98
vinyl acetate	43	86	dibromochloromethane	129	127, 131	2-ethylthiophene	97	45, 112
2-butanone	43	72	1,2-dibromoethane	107	109	1,2,3-trimethylbenzene	105	120
cis-1,2-dichloroethene	61	96, 98	butyl acetate	73	43, 56	indan	117	91, 118
chloroform	83	85, 47	octane	85	43, 57, 114	indene	115	89, 116
1,2-dichloroethane	62	49, 63, 61	tetrachloroethene	166	94, 131	1,2,4,5-tetramethylbenzene	119	91, 134
	29	43, 63, 61	1,1,1,2-tetrachloroethane	131	95, 133	benzothiophene	134	63, 89
acetaldehyde	41	43, 44	chlorobenzene	112	77, 114	2-methylnaphthalene	142	115, 141
isopentane		42, 57 57, 71	ethylbenzene	91	106	1-methylnaphthalene	142	115, 141
2-methylpentane 3-methylpentane	43 57	41, 56, 71	m+p-xylene	91	106	. mong map to broot	1.12	1

Table 7

## Table 8

# Internal Standards and the Associated Target Analytes

bromochic	promethane	1,4-difluorobenzene	chlorobenzene-D5			
chlorodifluoromethane	trans-1,2-dichloroethene	hexane	toluene	decane		
propylene	1,1-dichloroethane	diisopropyl ether	toluene-D8	benzyl chloride		
propane	МТВЕ	ethyl acetate	1,3-dichloropropane	1,3-dichlorobenzene		
dichlorodifluoromethane	vinyl acetate	2,2-dichloropropane	2-hexanone	1,4-dichlorobenzene		
chloromethane	2-butanone	tetrahydrofuran	dibromochloromethane	sec-butylbenzene		
Freon-114	cis-1,2-dichloroethene	tert-butyl ethyl ether	1,2-dibromoethane	p-isopropyltoluene		
methanol	chloroform	1,2-dichloroethane-D4	butyl acetate	1,2-dichlorobenzene		
vinyl chloride	1,2-dichloroethane	1,1,1-trichloroethane	octane	n-butylbenzene		
1,3-butadiene	trans-1,2-dichloroethene	1,1-dichloropropene	tetrachloroethene	1,2-dibromo-3- chloropropane		
butane	1,1-dichloroethane	benzene	1,1,1,2-tetrachloroethane	undecane		
bromomethane	MTBE	carbon tetrachloride	chlorobenzene	dodecane		
chloroethane	vinyl acetate	cyclohexane	ethylbenzene	1,2,4-trichlorobenzei		
dichlorofluoromethane	2-butanone	tert-amyl methyl ether	m+p-xylene	naphthalene		
ethanol	cis-1,2-dichloroethene	dibromomethane	bromoform	1,2,3-trichlorobenzei		
acetonitrile	chloroform	1,2-dichloropropane	styrene	hexachlorobutadiene		
vinyl bromide	1,2-dichloroethane	bromodichloromethane	1,1,2,2-tetrachloroethane	2-methylthiophene		
acrolein	acetaldehyde	trichloroethene	o-xylene	3-methylthiophene		
acetone	isopentane	1,4-dioxane	1,2,3-trichloropropane	2-ethylthiophene		
trichlorofluoromethane	2-methylpentane	2,2,4-trimethylpentane	nonane	1,2,3-trimethylbenze		
isopropyl alcohol	3-methylpentane	heptane	bromofluorobenzene	indan		
acrylonitrile		2,4,4-trimethyl-1-pentene	isopropylbenzene	indene 1,2,4,5-		
pentane		cis-1,3-dichloropropene	bromobenzene	tetramethylbenzene		
ethyl ether		4-methyl-2-pentanone	2-chlorotoluene	benzothiophene		
1,1-dichloroethene		2,4,4-trimethyl-2-pentene	n-propylbenzene	2-methylnaphthalen		
Tertiary butyl Alcohol		trans-1,3-dichloropropene	4-chlorotoluene	1-methylnaphthalen		
methylene chloride		1,1,2-trichloroethane	4-ethyl toluene			
3-chloropropene		thiophene	1,3,5-trimethylbenzene			
carbon disulfide			tert-butlybenzene			
Freon 113			1,2,4-trimethylbenzene			

Alpha Analytical, Inc. Technical Standard Operating Procedure **Air Analysis: TO-15** <u>Effective Date: May 22, 2009</u>

## Table 9A

## TO-15 Target Analytes and Reporting Limits Standard List

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m <sup>3</sup>	COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m <sup>3</sup>
1,1,1-trichloroethane	71-55-6	0.2	1.09	chloromethane	74-87-3	0.2	0.41
,1,2,2-tetrachloroethane	79-34-5	0.2	1.37	cis-1,2-dichloroethene	156-59-2	0.2	0.79
1,1,2-trichloroethane	79-00-5	0.2	1.09	cis-1,3-dichloropropene	10061-01-5	0.2	0.91
1,1-dichloroethane	75-34-3	0.2	0.81	cyclohexane	110-82-7	0.2	0.69
1,1-dichloroethene	75-35-5	0.2	0.79	dibromochloromethane	124-48-1	0.2	1.7
1,2,4-trichlorobenzene	120-82-1	0.2	1.48	dichlorodifluoromethane	75-71-8	0.2	0.99
1,2,4-trimethylbenzene	95-63-6	0.2	0.98	ethanol	64-17-5	2.5	4.71
1,2-dibromoethane	106-93-4	0.2	1.54	ethyl acetate	141-78-6	0.5	1.8
1,2-dichlorobenzene	95-50-1	0.2	1.2	ethylbenzene	100-41-4	0.2	0.87
1,2-dichloroethane	107-06-2	0.2	0.81	Freon-113	76-13-1	0.2	1.53
1,2-dichloropropane	78-87-5	0.2	0.92	Freon-114	76-14-2	0.2	1.4
1,3,5-trimethylbenzene	95-63-6	0.2	0.98	hexachlorobutadiene	87-68-3	0.2	2.13
1,3-butadiene	106-99-0	0.2	0.44	hexane	110-54-3	0.2	0.7
1,3-dichlorobenzene	541-73-1	0.2	1.2	isopropyl alcohol	67-63-0	0.5	1.23
1,4-dichlorobenzene	106-46-7	0.2	1.2	methylene chloride	75-09-2	0.5	1.74
1.4-dioxane	123-91-1	0.2	0.72	MIBK	108-10-1	0.2	0.82
2,2,4-trimethylpentane	540-84-1	0.2	0.93	MTBE	1634-04-4	0.2	0.72
2-butanone	78-93-3	0.2	0.59	m+p-xylene	108-38-3 106-42-3	0.4	1.74
2-hexanone	591-78-6	0.2	0.82	n-heptane	142-82-5	0.2	0.82
3-chloropropene	107-05-1	0.2	0.63	o-xylene	95-47-6	0.2	0.87
4-Ethyltoluene	622-96-8	0.2	0.98	propylene	115-7-1	0.2	0.34
Acetone	67-64-1	0.2	0.47	styrene	100-42-5	0.2	0.85
benzene	71-43-2	0.2	0.64	tetrachloroethene	127-18-4	0.2	1.36
Benzyl Chloride	100-44-7	0.2	1.03	tetrahydrofuran	109-99-9	0.2	0.59
bromodichloromethane	75-27-4	0.2	1.34	toluene	108-88-3	0.2	0.75
bromoform	75-25-2	0.2	2.07	trans-1,2- dichloroethene trans-1,3-	156-60-5	0.2	0.79
bromomethane	74-83-9	0.2	0.78	dichloropropene	10061-02-6	0.2	0.79
carbon disulfide	75-15-0	0.2	0.62	trichloroethene	79-01-6	0.2	0.91
carbon tetrachloride	56-23-5	0.2	1.26	trichlorofluoromethane	75-69-4	0.2	1.07
chlorobenzene	108-90-7	0.2	0.92	vinyl acetate	108-05-4	0.2	1.12
chloroethane	75-00-3	0.2	0.53	vinyl bromide	593-60-2	0.2	0.7
chloroform	67-66-3	0.2	0.98	vinyl chloride	75-01-4	0.2	0.87

### Table 9B

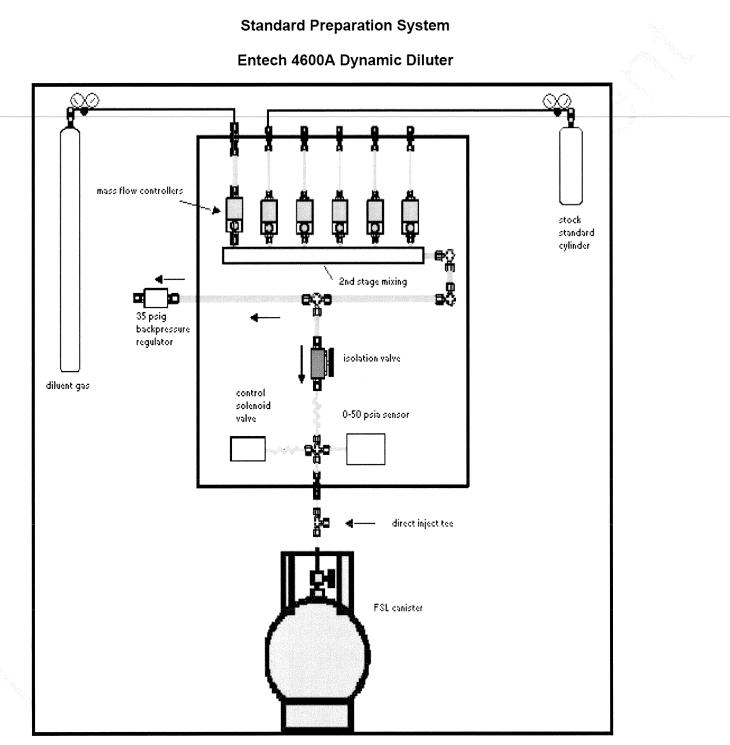
# TO-15 Target Analytes and Reporting Limits Additional Analytes

r

COMPOUND	CAS#	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m <sup>3</sup>
A	P-42 Anal	ytes	
acrolein	107-02-8	0.20	0.46
acrylonitrile	107-13-1	0.20	0.43
butane	106-97-8	0.20	0.48
Chlorodifluoromethane	75-45-6	0.20	0.71
Dichlorofluoromethane	75-71-8	0.20	0.84
n-Pentane	109-66-0	0.20	0.59
Propane	74-98-6	0.20	0.36
MADEP	MCP 826	0 Analytes	
1,1,1,2-tetrachloroethene	630-20-6	0.20	1.37
1,1-dichloropropene	563-58-6	0.20	0.91
1,2,3-trichlorobenzene	87-61-6	0.20	1.48
1,2,3-Trichloropropane	96-18-4	0.20	1.20
1,3-dichloropropane	142-28-9	0.20	0.92
2,2-dichloropropane	594-20-7	0.20	0.92
2-chlorotoluene	95-49-8	0.20	1.03
4-chlorotoluene	106-43-4	0.20	1.03
bromobenzene	108-86-1	0.20	0.79
1,2-dibromo-3- chloropropane	96-12-8	0.20	1.93
dibromomethane	74-95-3	0.20	1.42
diisopropyl ether	108-20-3	0.20	0.84
isopropylbenzene	98-82-8	0.20	0.98
isopropyltoluene	99-87-6	0.20	1.10
naphthalene	91-20-3	0.20	1.05
n-butylbenzene	104-51-8	0.20	1.10
n-propylbenzene	103-65-1	0.20	0.98
sec-butylbenzene	135-98-8	0.20	1.10
tert-amyl methyl ether	994-05-8	0.20	0.84
tert-Butyl ethyl ether	637-92-3	0.20	0.84
tert-butylbenzene	98-06-6	0.20	1.10

COMPOUND	CAS #	Standard Reporting Limit, ppbV	Standard Reporting Limit, ug/m <sup>3</sup>
NYDEC Petro	eum Indic	ator Compo	unds
nonane	111-84-2	0.20	1.05
octane	111-65-9	0.20	0.93
undecane	1120-21-4	0.20	1.28
decane	124-18-5	0.20	1.16
dodecane	112-40-3	0.20	1.39
indene	95-13-6	0.20	0.95
Indan	496-11-7	0.20	0.97
thiophene	110-02-1	0.20	0.69
2-methylthiophene	554-13-3	0.20	0.80
3-methylthiophene	616-44-4	0.20	0.80
2-ethyl thiophene	872-55-9	0.20	0.92
benzothiophene	934-80-5	2.5	13.7
1,2,3-trimethylbenzene	526-73-8	0.20	0.98
1,2,4,5- tetramethylbenzene	95-93-2	2.5	13.7
2-methylnaphthalene	91-57-6	2.5	14.5
1-methylnaphthalene	90-12-0	2.5	14.5
Projec	t Specific	Analytes	
acetaldehyde	75-07-0	2.5	0.36
Acetonitrile	75-05-8	0.20	0.34
butyl acetate	123-86-4	0.50	0.95
ethyl ether	60-29-7	0.30	0.93
methanol	67-56-1	5.0	6,55
2,4,4-trimethyl-1-pentene	107-39-1	0.50	0.33 2.29
2,4,4-trimethyl-2-pentene	107-39-1	0.50	2.29
tert-butyl alcohol	75-65-0	0.30	0.61
isopentane	78-78-4	0.20	0.59
2-methylpentane	107-83-5	0.20	0.39
3-methylpentane	96-14-0	0.20	0.70
- s mourypontano	00-17-0	0.20	0.10

# Figure 3



#### Appendix A

#### Cold Trap Dehydration technique (CTD) for Analysis of Sulfides and Mercaptans by EPA TO-15

Target analytes:

Compound	CAS#	Reporting Limit, ppbV
Hydrogen sulfide	7783-06-4	5.0
Carbonyl sulfide	463-58-1	1.0
Methyl mercaptan	74-93-1	5.0
Ethyl mercaptan	75-08-1	1.0
Dimethyl sulfide	75-18-3	1.0
carbon disulfide	75-15-0	0.5
Isopropyl Mercaptan	75-33-2	2.5
tert-Butyl Mercaptan	75-66-1	2.5
n-Propyl Mercaptan	107-03-9	1.0
Ethyl Methyl Sulfide	624-89-5	0.5
	-9. 	

Compound	CAS#	Reporting Limit, ppbV
Thiophene	110-02-1	0.5
Isobutyl Mercaptan	513-44-0	5.0
diethyl sulfide	352-93-2	0.5
Butyl Mercaptan	109-75-5	5.0
dimethyl disulfide	624-92-0	0.5
3-Methylthiophene	616-44-4	0.5
Tetrahydrothiophene	110-01-0	2.0
2-Ethylthiophene	872-55-9	0.5
2,5-Dimethylthiophene	638-02-8	0.5
Diethyl Disulfide	110-81-6	1.0

The cold trap dehydration method requires removal of the glass bead trap installed in module 1 of the Entech 7100A concentrator and installing a blank trap (i.e. no trapping material). This trap is cooled and a 250 mL aliquot of sample is allowed to pass through this trap and then directly onto the Tenax trap in module 2, which is also cooled (see Table A-4 for setpoints). The sample is then transferred to module 3 (cryofocusser) via ballistic heating. All requirements stated in this SOP must be applied to any TO15-Sulfide/Mercaptan analysis conducted in the laboratory.

SOP modifications:

Standard preparation and calibration procedures for these analytes are listed in Table A-1, A-2, and Table A-3. Quantitation parameters are listed in Table A-5.

Section 9.2.5 and 9.5.3.1: Use the Entech method alpha\_H2S&SULF.CTD in place of the alpha\_TO15.MPT method.

A second source laboratory check standard (LCS) is not readily available for these analytes. Analyze a continuing calibration standard to verify initial calibration.

#### TO15-CTD Tedlar® Bag Stock Standard Preparation

COMPOUND (liquids)	MOL WGT	Density ug/uL	uL injected	FINAL ppmV
ethyl mercaptan	62.14	839	6.2	100
dimethyl sulfide	62.14	846	6.1	100
carbon disulfide	76.1	1266	5.0	100
Isopropyl Mercaptan	76.2	820	7.7	100
tert-Butyl Mercaptan	90.19	800	9.4	100
n-Propyl Mercaptan	76.16	841	7.5	100
Ethyl Methyl Sulfide	76.16	842	7.5	100
Thiophene S.G.	84.1	1051	6.7	101
Isobutyl Mercaptan	90.19	831	9.0	100
diethyl sulfide	90.2	837	9.0	100
Butyl Mercaptan	90.19	842	8.9	100
dimethyl disulfide	94.2	1046	7.5	100
3-Methylthiophene	98.17	1016	8.0	100
Tetrahydrothiophene	88.17	1000	7.3	100
2-Ethylthiophene	112.19	990	9.4	100
2,5-Dimethylthiophene	112.19	985	9.5	100
Diethyl Disulfide	122.25	993	10.2	100
COMPOUND (gases)	MOL WGT	CONC ppmV	mL injected	Conc ppmV
hydrogen sulfide	34.08	1.00E+06	2.0	100.0
carbonyl sulfide	60.08	1.00E+06	2.0	100.0
methyl mercaptan	48.11	1.00E+06	2.0	100.0

### Summary of Secondary Standards Preparation for Sulfides and Mercaptan Analysis

Primary Standard	Primary Standard Conc. ppmV	Volume of Primary Standard Injected into canister	Primary Standard Transfer Data	Final Volume Tedlar® Bag (L)	Final Concentration ppbV
Secondary standards prepared using gas-tight syringes					
Tedlar® bag primary standard	100	0.16 mL	Syringe Injection	4.0	100
Tedlar® bag primary standard	100	4.0 mL	Syringe Injection	4.0	4.0

#### Table A-3

#### **Calibration Standard Levels**

	where Weak Strategies and	
Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.40	25 mL_of 4.0 ppbV sec. standard
2	0.80	50 mL_of 4.0 ppb∨ sec. standard
3	4.0	250 mL_of 4.0 ppbV sec. standard
4	10	25 mL_of 100 ppbV sec. standard
5	20	50 mL_of 100 ppb∀ sec. standard
6	50	125 mL_of 100 ppbV sec. standard
<u> </u>	80	200 mL_of 100 ppb∀ sec. standard
8	100	250 mL_of 100 ppbV sec. standard

# ENTECH 7016CA/7100A Operating Parameters for CTD Method

Module 1 (Glass Bead Trap)			
Parameter	Setting		
Trapping Temperature	-20° C		
Internal standard volume	25 mL		
Internal standard flow rate	60 mL / min		
Sample volume (may vary depending on sample concentrations)	250 mL		
Sample flow rate	60 mL / min		
Preheat Temperature	10º C		
Desorb Temperature	10º C		
Bake Temperature	150° C		
Bake Time	7 min.		
Module 2 (Tenax t	rap)		
Parameter	Setting		
Trapping Temperature	80 <sup>0</sup> C		
Desorb Temperature	180° C		
Bake Temperature	190° C		
Module 2 to Module 3 desorb time	3 min		
Module 3 (Cryofocu	sser)		
Parameter	Setting		
Cryofocusing Temperature	-150° C		
Desorb Temperature	Approx. 70° C		
Module 3 to GC desorb time	2 min		
Bake temperature / event	Approx. 90° C / event 3		
Delay time	20 min		

# Internal Standard (IS) Assignments and Quantitation lons for Sulfides & Mercaptans

	Quant	Sec.
Compound	lon	lon(s)
Bromochloromethane (IS)	49	130
Hydrogen sulfide	34	33, 36
Carbonyl sulfide	60	62, 32
Methyl mercaptan	47	48, 45
ethyl mercaptan	62	47, 45
dimethyl sulfide	62	61, 47
carbon disulfide	76	44, 78
Isopropyl Mercaptan	76	43, 61
tert-Butyl Mercaptan	57	41, 90
n-Propyl Mercaptan	76	47, 41
Ethyl Methyl Sulfide	61	76, 48

Compound	Quant Ion	Sec. lon(s)
1,4-difluorobenzene (IS)	114	63
Thiophene	84	58, 45
Isobutyl Mercaptan	41	56, 90
diethyl sulfide	75	90, 47
Butyl Mercaptan	56	41, 90
dimethyl disulfide	94	79, 45
		i. Livi
	. 1	

Compound	Quant	Sec.
Compound	lon	lon(s)
Chlorobenzene-D5 (IS)	54	82, 117
3-Methylthiophene	97	98, 45
Tetrahydrothiophene	60	88, 45
2-Ethylthiophene	97	98, 45
2,5-Dimethylthiophene	111	112,97
Diethyl Disulfide	122	66, 94
Toluene-D8	98	100
Bromofluorobenzene	95	174

# Appendix B

#### Data Acquistion Parameters and Analysis Modifications for Conducting SIM Analysis

SIM analysis is conducted when full scan sensitivity does not meet the data quality objectives (DQO) of the project and/or regulatory criteria. The acquisition method used to acquire full scan data simultaneously acquires SIM data using the SIM ions and windows specified in Table B-1. The following modifications to the full scan SOP must be done to generate data using the SIM signal:

- SIM level calibration standards must be analyzed w/ the full scan curve (0.02 and 0.04 ppbV)
- A calibration curve is generated using the SIM signal utilizing the lower level calibration standards and must meet the same criteria as the full scan calibration criteria.
- The continuing calibration and/or LCS should be analyzed at a lower concentration (5.0 ppbV)
- Method blanks must be evaluated for the SIM reporting limit as listed in Table B-2.

The SIM signal only acquires data for a limited target analyte list. These target analytes and reporting limits are listed in Table B-2. All requirements stated in this SOP must be applied to any TO15-SIM analysis conducted in the laboratory.

# Table B-1

#### Calibration Standard Levels for SIM Analysis

Calibration Level	Amount (ppbV)	Volume / Secondary Standard
1	0.02	125 mL_of 0.04 ppb∀ sec. standard
2	0.04	250 mL_of 0.04 ppb∀ sec. standard
3	0.20	250 mL of 0.2 ppb∀ sec. standard
4	0.50	125 mL of 1.0 ppb∀ sec. standard
5	1.0	250 mL of 1.0 ppbV sec. standard
6	2.5	125 mL_of 5.0 ppb∀ sec. standard
7	5.0	250 mL of 5.0 ppbV sec. standard
8	10	125 mL of 20 ppbV sec. standard
9	20	250 mL of 20 ppbV sec. standard
10	50	125 mL_of 100 ppbV sec. standard

# Table B-2

# Seletive Ion Monitoring (SIM) Groupings

Group ID Start Time (may vary)	Compounds in Group	lons Scanned	Group ID Start Time (may vary)	Compounds in Group	lons Scanned
Start Time (may vary)	Dichlorodifluoromethane		Start Time (may vary)		
Auto_2 4.00 min	Chloromethane Freon-114 vinyl chloride 1,3-butadiene	39, 50, 52, 54, 62, 64, 85, 87, 135	Auto-16 12.20 min	benzene carbon tetrachloride 1,4-difluorobenzene	52, 63, 78, 82, 114, 117, 119
Auto_3 6.00 min	bromomethane	94, 96	Auto-17 13.00 min	1,2-dichloropropane bromodichloromethane trichloroethene	39, 62, 63, 83, 85, 97, 129, 130, 132
Auto_4 6.25 min	chloroethane	64, 66	Auto-18 13.80 min	cis-1,3-dichloropropene 4-methyl-2-pentanone	39, 43, 58, 75, 77, 100
Auto_5 6.90 min	Acetone	43, 58	Auto-19 14.75 min	trans-1,3-dichloropropene 1,1,2-trichloroethane Toluene Toluene-D8	39, 61, 75, 77, 83, 91, 92, 97, 98, 100
Auto_6 7.25 min	trichlorofluoromethane	101, 103	Auto-20 15.50 min	Dibromochloromethane	127, 129, 131
Auto_7 7.50 min	Acrylonitrile	51, 52, 53	Auto-21 15.95 min	1,2-dibromoethane	107, 109
Auto_8 7.90 min	1,1-dichloroethene methylene chloride	49, 61, 63, 84, 96	Auto-22 16.20 min	Tetrachloroethene	94, 131, 166
Auto_9 8.40 min	Freon-113	85, 101, 151	Auto-23 16.60 min	1,1,1,2-tetrachloroethane Chlorobenzene-D5 chlorobenzene	54, 77, 82, 95, 112, 114, 117, 131, 133
Auto-10 9.00 min	trans-1,2-dichloroethene Halothane	61, 96, 98, 117, 198	Auto-24 17.09 min	ethylbenzene p & m-xylene bromoform styrene 1,1,2,2-tetrachloroethane o-xylene	78, 83, 85, 91, 103, 104, 106, 171, 173, 175
Auto-11 9.45 min	1,1-dichloroethane MTBE	43, 57, 63, 65, 73	Auto-25 17.95 min	Bromofluorobenzene isopropylbenzene	75, 95, 105, 120, 174
Auto-12 9.85 min	2-butanone	43, 72	Auto-26 18.30	4-ethyl toluene 1,3,5-trimethylbenzene	91, 105, 120
Auto-13 10.4 min	cis-1,2-dichloroethene bromochloromethane chloroform	47, 49, 61, 83, 85, 96, 98, 130	Auto-27 18.85 min	1,2,4-trimethylbenzene dichlorobenzene isomers sec-butylbenzene p-isopropyltoluene n-butylbenzene	75, 91, 105, 111, 119, 120, 134, 146
Auto-14 11.00 min	1,2-dichloroethane 1,2-dichloroethane-D4	49, 61, 62, 63, 65, 67, 102	Auto-28 20.27 min	naphthalene 1,2,4-trichlorobenzene 1,2,3-trichlorobenzene hexachlorobutadiene	102, 109, 118, 128, 145, 180, 225, 260
Auto-15 11.70 min	1,1,1-trichloroethane	61, 97, 119			

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# Table B-3

	F	¥	· · · · · · · · · · · · · · · · · · ·		-	-	-
COMPOUND	CAS #	SIM Reporting Limit, ppbV	SIM Reporting Limit, ug/m <sup>3</sup>	COMPOUND	CAS #	SIM Reporting Limit, ppbV	SIM Reporting Limit, ug/m <sup>3</sup>
1,1,1-trichloroethane	71-55-6	0.020	0.109	Freon-114	76-14-2	0.050	0.349
1,1,2,2- tetrachloroethane	79-34-5	0.020	0.137	methylene chloride	75-09-2	0.500	1.74
1,1,2-trichloroethane	79-00-5	0.020	0.109	MTBE	1634-04-4	0.020	0.072
1,1-dichloroethane	75-34-3	0.020	0.081	m+p-xylene	108-38-3 106-42-3	0.040	0.174
1,1-dichloroethene	75-35-5	0.020	0.079	o-xylene	95-47-6	0.020	0.087
1,2,4-trimethylbenzene	95-63-6	0.020	0.098	styrene	100-42-5	0.020	0.085
1,2-dibromoethane	106-93-4	0.020	0.154	tetrachloroethene	127-18-4	0.020	0.136
1,2-dichlorobenzene	95-50-1	0.020	0.120	toluene	108-88-3	0.050	0.188
1,2-dichloroethane	107-06-2	0.020	0.081	trans-1,2- dichloroethene trans-1,3-	156-60-5	0.020	0.079
1,2-dichloropropane	78-87-5	0.020	0.092	dichloropropene	10061-02-6	0.020	0.091
1,3,5-trimethylbenzene	95-63-6	0.020	0.098	trichloroethene	79-01-6	0.020	0.107
1,3-butadiene	106-99-0	0.020	0.044	1,2,4-trichlorobenzene	120-82-1	0.050	0.371
1,3-dichlorobenzene	541-73-1	0.020	0.120	trichlorofluoromethane	75-69-4	0.050	0.281
1,4-dichlorobenzene	106-46-7	0.020	0.120	vinyl chloride	75-01-4	0.020	0.051
benzene	71-43-2	0.070	0.223	CT RS	R Addition	al Analytes	
bromodichloromethane	75-27-4	0.020	0.134	1,1,1,2- tetrachloroethane	630-20-6	0.020	0.137
bromoform	75-25-2	0.020	0.207	acrylonitrile	107-13-1	0.500	1.08
bromomethane	74-83-9	0.020	0.078	isopropyltoluene	99-87-6	0.500	2.74
carbon tetrachloride	56-23-5	0.020	0.126	n-butylbenzene	104-51-8	0.500	2.74
chlorobenzene	108-90-7	0.020	0.092	sec-butylbenzene	135-98-8	0.500	2.74
chloroethane	75-00-3	0.020	0.053	tert-butylbenzene	98-06-6	0.500	2.74
chloroform	67-66-3	0.020	0.098	isopropylbenzene	98-82-8	0.500	2.46
chloromethane	74-87-3	0.500	1.03	2-butanone (MEK)	78-93-3	0.500	1.48
cis-1,2-dichloroethene	156-59-2	0.020	0.079	Acetone	67-64-1	2.000	4.70
cis-1,3-dichloropropene	10061-01- 5	0.020	0.091	2-methyl-2-pentanone (MIBK)	108-10-1	0.500	2.05
dibromochloromethane	124-48-1	0.020	0.170	Proje	ect Specific	Analytes	
dichlorodifluoromethane	75-71-8	0.050	0.247	Naphthalene	91-20-3	0.050	0.262
ethylbenzene	100-41-4	0.020	0.087	Halothane	151-67-7	0.120	0.968
Freon-113	76-13-1	0.050	0.383	1,2,3-trichlorobenzene	87-61-6	0.140	1.04

# TO15-SIM Target Analytes and Reporting Limits

# Appendix D

Community Air Monitoring Plan



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# Appendix D Community Air Monitoring Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

March 7, 2011

Christopher D. Keen Senior Scientist

, Feldman

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#### Appendix D Community Air Monitoring Plan

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

Prepared for: National Grid

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Our Ref.: B0036704.0001.00101

Date: March 7, 2011

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#### Attachments

- D-1 NYSDOH Generic Community Air Monitoring Plan
- D-2 NYSDEC TAGM #4031 Fugitive Dust Suppression and Particulate Monitoring Program at Inactive Hazardous Waste Sites

## Appendix D Community Air Monitoring Plan

Former Dangman Park Manufactured Gas Plant Site

#### 1. Introduction

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this Community Air Monitoring Plan (CAMP) as a component of the Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York. The CAMP fulfills the general requirements set forth by the New York State Department of Health (NYSDOH) Generic Community Air Monitoring Plan (Attachment D-1 of this CAMP) and the New York State Department of Environmental Conservation (NYSDEC) Technical and Administrative Guidance Memorandum (TAGM) #4031 "Fugitive Dust Suppression and Particulate Monitoring Program at Inactive Hazardous Waste Sites" (Attachment D-2 of this CAMP). The intent of this CAMP is to provide procedures to protect the downwind communities from potential airborne releases of constituents of concern during RI activities. As such, this CAMP specifies the potential air emissions, air monitoring procedures, monitoring schedule, and data collection and reporting for the RI activities to be conducted.

The CAMP should be used in conjunction with the RI Work Plan, the Field Sampling Plan (FSP), the Quality Assurance Project Plan (QAPP), and the Health and Safety Plan (HASP). The RI Work Plan presents the Site background and defines the field sampling program. The FSP describes the methods and procedures to be used for environmental sample collection during implementation of the RI field activities. The QAPP presents the quality assurance/quality control (QA/QC) procedures to be used during implementation of the RI Work Plan, as well as a description of the general field and laboratory procedures. The FSP, QAPP, and HASP are provided as Appendices B, C, and E of the RI Work Plan, respectively.

#### 1.1 Site Description

The Site is located at 486 Neptune Avenue in the Borough of Brooklyn, New York City, Kings County, New York and occupies portions of two parcels that are identified by Tax Map Number: Block 7273, Lots 1 and 25. As shown on Figure 1 of the RI Work Plan, the Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is generally flat with an elevation of approximately 9 feet above mean sea level (msl). The closest natural surface water body is Coney Island Creek, which is located approximately 0.25 miles to the northwest of the Site.



Former Dangman Park Manufactured Gas Plant Site

The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5<sup>th</sup> Street to the east, a residential parcel to the south, and a commercial parcel to the west. Currently, the Site is developed with a shopping center and a parking lot for a high-rise apartment building.

#### 1.2 Summary of Site Investigation Activities

The proposed RI activities for the Site include subsurface soil sampling, monitoring well drilling, installation, and development, groundwater sampling, and soil vapor sampling. A more detailed description of the investigation activities can be found in the RI Work Plan.

#### 1.3 Potential Air Emissions Related to Investigation Activities

Certain intrusive RI activities to be conducted at the Site have the potential to generate localized impacts to air quality including drilling and subsurface soil sampling. Some non-intrusive RI activities to be conducted also have the potential to generate localized impacts to air quality and include the collection of groundwater samples.

#### 1.4 Air/Odor Emissions and Control Measures

Air emissions control and fugitive dust suppression techniques will be used during the RI activities identified above, as necessary, to limit the air/odor emissions from the Site. Air monitoring will be conducted during both intrusive and non-intrusive Site activities.

Odor and dust control measures will be available at the Site and used when necessary. The following dust and odor suppression measures may be used during the RI activities, depending upon specific circumstances and air monitoring results:

- Water spray
- Polyethylene sheeting (for covering drill cuttings, soil stockpiles, etc.)
- Containerize drill cuttings and groundwater in 55-gallon drums with the cover secured



Former Dangman Park Manufactured Gas Plant Site

Odor and dust control measures will be implemented based on visual or olfactory observations, and the results of airborne particulate and volatile organic compound (VOC) monitoring.

#### 2. Air Monitoring Procedures

Real-time air monitoring will be implemented at the Site for VOCs and particulate matter. Particulate monitoring will not be performed, however, during non-intrusive activities (i.e., groundwater sampling) and precipitation events. Upwind and downwind monitoring locations will be determined through visual observation (windsock or similar technique). Monitoring at each location will include the use of hand-held direct-reading survey instruments.

#### 2.1 Monitoring Location Selection

Monitoring locations will be determined daily based on visual observation of a wind direction. A single upwind (background) location will be selected daily where both VOC and particulate monitoring will be conducted. This upwind location will be established at the start of each day before commencing investigation activities. Monitoring activities will continue at a single downwind location throughout the day. If wind direction shifts radically during the day (greater than approximately +/- 60 degrees from original upwind) new upwind and downwind monitoring locations will be established. Any location changes will be documented in the field logbook.

#### 2.2 VOCs and PAHs Monitoring

As required by the NYSDOH guidance for community air monitoring during intrusive activities, VOCs will be monitored continuously during intrusive Site activities (drilling of soil borings or installation of monitoring wells) with instrumentation that is equipped with electronic data-logging capabilities. Because real-time monitors for polycyclic aromatic hydrocarbons (PAHs) do not exist, the real-time VOC monitoring equipment will also serve as surrogate monitoring equipment for PAH emissions at the Site. A photoionization detector (PID) (MiniRAE 2000 [or equivalent]) will be used to conduct the real-time VOC and PAH monitoring. All 15-minute readings will be recorded by the equipment's electronic data-logging system; any instantaneous readings collected to facilitate activity decisions will be recorded in the field logbook.

During non-intrusive Site activities (monitoring well development and collection of groundwater samples from monitoring wells), periodic VOC monitoring will be



Former Dangman Park Manufactured Gas Plant Site

conducted. Periodic monitoring may include monitoring upon arrival at the sample location, while opening a well cap, during well bailing and/or purging, and/or prior to leaving a sample location. However, if a sampling location is proximal to potentially exposed individuals, VOCs will be monitored continuously during sampling activities at that location.

#### 2.3 Particulate Matter Monitoring

As required by the NYSDOH guidance, particulate matter will be monitored continuously during intrusive Site activities (drilling of soil borings or installation of monitoring wells) with instrumentation that is equipped with electronic data-logging capabilities. A particulate monitor (Thermo Scientific personal DataRAM pDR-1500 [or equivalent]) will be used to conduct the real-time monitoring of particulate matter less than 10 microns in size (PM-10). All 15-minute readings will be recorded by the equipment's electronic data-logging system; any instantaneous readings collected to facilitate activity decisions will be recorded in the field logbook.

Fugitive dust migration will be visually assessed during all work activities, and reasonable dust suppression techniques will be used during any Site activities that may generate fugitive dust. Fugitive dust control measures are discussed in Section 1.4 of this CAMP.

#### 2.4 Action Levels

The action levels provided below are to be used to initiate response actions, if necessary, based on real-time monitoring.

#### 2.4.1 Action Levels for VOCs and PAHs

As outlined in the NYSDOH CAMP guidance document, if the ambient air concentration of total VOCs exceeds 5 parts per million (ppm) above background (upwind location) for the 15-minute average, intrusive Site activities will be temporarily halted while monitoring continues. If the total VOC concentration readily decreases (through observation of instantaneous readings) below 5 ppm above background, then intrusive Site activities can resume with continuous monitoring.

If the ambient air concentrations of total VOCs persist at levels in excess of 5 ppm above background but are less than 25 ppm above background, intrusive Site activities will be halted, the source of the elevated VOC concentrations will be identified,



Former Dangman Park Manufactured Gas Plant Site

corrective actions to reduce or abate the emissions will be undertaken, and air monitoring will be continued. Once these actions have been implemented, intrusive Site activities can resume provided that the following two conditions are met:

- The 15-minute average VOC concentrations remain below 5 ppm above background.
- The total VOC concentration 200 feet downwind of the sample location or half the distance to the nearest potential receptor or residential/commercial structure (whichever is less but in no case less than 20 feet) is below 5 ppm above background for the 15-minute average.

If the ambient air concentrations of total VOCs exceed 25 ppm above background, the intrusive Site activities must cease, and emissions control measures must be implemented.

Periodic monitoring for VOCs is required during non-intrusive activities. During these activities, ambient direct-reading (instantaneous) VOC data will be periodically collected at the location of the non-intrusive activity and recorded in the field activity logbooks.

#### 2.4.2 Action Levels for PM-10

As required by the NYSDOH guidance, if the ambient air concentration of PM-10 at any one (or more) of the monitoring locations is noted at levels in excess of 100 micrograms per cubic meter ( $\mu$ g/m<sup>3</sup>) above background (upwind location), or if airborne dust is observed leaving the work area, intrusive Site activities will be temporarily halted. The source of the elevated PM-10 concentration will be identified, corrective actions to reduce or abate the emissions will be undertaken, and air monitoring will be continued. Work may continue following the implementation of dust suppression techniques provided the PM-10 levels do not exceed 150  $\mu$ g/m<sup>3</sup> above background.

If, after implementation of dust suppression techniques, PM-10 levels are greater than  $150 \ \mu g/m^3$  above background, work must be stopped and Site activities must be reevaluated. Work may only resume provided that the dust suppression measures and other controls are successful in reducing PM-10 levels to less than  $150 \ \mu g/m^3$  above background and in preventing visible dust from leaving the Site.



Former Dangman Park Manufactured Gas Plant Site

If the ambient air concentration of PM-10 is above 150  $\mu$ g/m<sup>3</sup> above background, the intrusive Site activities must cease and emissions control measures must be implemented.

#### 2.5 Meteorological Monitoring

Wind direction is the only meteorological information considered relevant for the RI activities and CAMP. Meteorological monitoring will be conducted periodically at the Site using a windsock or other appropriate equipment. Wind direction will be established at the start of each work day and may be re-established at any time during the work day if a significant shift in wind direction is noted.

#### 2.6 Instrument Calibration

Calibration of the VOC (PID) and PM-10 (particulate monitor) monitoring instrumentation will be performed in accordance with each of the equipment manufacturer's calibration and quality assurance requirements. The VOC and PM-10 monitoring instrumentation will be calibrated at least daily, and calibrations will be recorded in the field activity logbook.

#### 3. Monitoring Schedule and Data Collection and Reporting

This section presents the monitoring schedule and data collection and reporting requirements.

#### 3.1 Monitoring Schedule

Real-time VOC and PM-10 monitoring will be performed continuously throughout the intrusive activities. VOC monitoring will be performed periodically during non-intrusive sampling-type activities. Wind direction will be determined at the start of each day and at any other appropriate time during RI activities.

#### 3.2 Data Collection and Reporting

Air monitoring data will be collected continuously by the VOC and PM-10 monitoring equipment during intrusive Site activities by an electronic data-logging system. The data management software will be set up so that instantaneous observed readings are recorded by the electronic data acquisition system and averaged over 15-minute time



Former Dangman Park Manufactured Gas Plant Site

periods. All readings will be recorded and archived, and will be available for review by NYSDOH and NYSDEC personnel.

# Attachment D-1

NYSDOH Generic Community Air Monitoring Plan

## Appendix 1A New York State Department of Health Generic Community Air Monitoring Plan

# Overview

A Community Air Monitoring Plan (CAMP) requires real-time monitoring for volatile organic compounds (VOCs) and particulates (i.e., dust) at the downwind perimeter of each designated work area when certain activities are in progress at contaminated sites. The CAMP is not intended for use in establishing action levels for worker respiratory protection. Rather, its intent is to provide a measure of protection for the downwind community (i.e., off-site receptors including residences and businesses and on-site workers not directly involved with the subject work activities) from potential airborne contaminant releases as a direct result of investigative and remedial work activities. The action levels specified herein require increased monitoring, corrective actions to abate emissions, and/or work shutdown. Additionally, the CAMP helps to confirm that work activities did not spread contamination off-site through the air.

The generic CAMP presented below will be sufficient to cover many, if not most, sites. Specific requirements should be reviewed for each situation in consultation with NYSDOH to ensure proper applicability. In some cases, a separate site-specific CAMP or supplement may be required. Depending upon the nature of contamination, chemical- specific monitoring with appropriately-sensitive methods may be required. Depending upon the proximity of potentially exposed individuals, more stringent monitoring or response levels than those presented below may be required. Special requirements will be necessary for work within 20 feet of potentially exposed individuals or structures and for indoor work with co-located residences or facilities. These requirements should be determined in consultation with NYSDOH.

Reliance on the CAMP should not preclude simple, common-sense measures to keep VOCs, dust, and odors at a minimum around the work areas.

### Community Air Monitoring Plan

Depending upon the nature of known or potential contaminants at each site, real-time air monitoring for VOCs and/or particulate levels at the perimeter of the exclusion zone or work area will be necessary. Most sites will involve VOC and particulate monitoring; sites known to be contaminated with heavy metals alone may only require particulate monitoring. If radiological contamination is a concern, additional monitoring requirements may be necessary per consultation with appropriate DEC/NYSDOH staff.

**Continuous monitoring** will be required for all <u>ground intrusive</u> activities and during the demolition of contaminated or potentially contaminated structures. Ground intrusive activities include, but are not limited to, soil/waste excavation and handling, test pitting or trenching, and the installation of soil borings or monitoring wells.

**Periodic monitoring** for VOCs will be required during <u>non-intrusive</u> activities such as the collection of soil and sediment samples or the collection of groundwater samples from existing monitoring wells. "Periodic" monitoring during sample collection might reasonably consist of taking a reading upon arrival at a sample location, monitoring while opening a well cap or

overturning soil, monitoring during well baling/purging, and taking a reading prior to leaving a sample location. In some instances, depending upon the proximity of potentially exposed individuals, continuous monitoring may be required during sampling activities. Examples of such situations include groundwater sampling at wells on the curb of a busy urban street, in the midst of a public park, or adjacent to a school or residence.

# VOC Monitoring, Response Levels, and Actions

Volatile organic compounds (VOCs) must be monitored at the downwind perimeter of the immediate work area (i.e., the exclusion zone) on a continuous basis or as otherwise specified. Upwind concentrations should be measured at the start of each workday and periodically thereafter to establish background conditions, particularly if wind direction changes. The monitoring work should be performed using equipment appropriate to measure the types of contaminants known or suspected to be present. The equipment should be calibrated at least daily for the contaminant(s) of concern or for an appropriate surrogate. The equipment should be capable of calculating 15-minute running average concentrations, which will be compared to the levels specified below.

1. If the ambient air concentration of total organic vapors at the downwind perimeter of the work area or exclusion zone exceeds 5 parts per million (ppm) above background for the 15-minute average, work activities must be temporarily halted and monitoring continued. If the total organic vapor level readily decreases (per instantaneous readings) below 5 ppm over background, work activities can resume with continued monitoring.

2. If total organic vapor levels at the downwind perimeter of the work area or exclusion zone persist at levels in excess of 5 ppm over background but less than 25 ppm, work activities must be halted, the source of vapors identified, corrective actions taken to abate emissions, and monitoring continued. After these steps, work activities can resume provided that the total organic vapor level 200 feet downwind of the exclusion zone or half the distance to the nearest potential receptor or residential/commercial structure, whichever is less - but in no case less than 20 feet, is below 5 ppm over background for the 15-minute average.

3. If the organic vapor level is above 25 ppm at the perimeter of the work area, activities must be shutdown.

4. All 15-minute readings must be recorded and be available for State (DEC and NYSDOH) personnel to review. Instantaneous readings, if any, used for decision purposes should also be recorded.

# Particulate Monitoring, Response Levels, and Actions

Particulate concentrations should be monitored continuously at the upwind and downwind perimeters of the exclusion zone at temporary particulate monitoring stations. The particulate monitoring should be performed using real-time monitoring equipment capable of measuring particulate matter less than 10 micrometers in size (PM-10) and capable of integrating over a period of 15 minutes (or less) for comparison to the airborne particulate action level. The equipment must be equipped with an audible alarm to indicate exceedance of the action level. In addition, fugitive dust migration should be visually assessed during all work activities.

1. If the downwind PM-10 particulate level is 100 micrograms per cubic meter  $(mcg/m^3)$  greater than background (upwind perimeter) for the 15-minute period or if airborne dust is observed leaving the work area, then dust suppression techniques must be employed. Work may continue with dust suppression techniques provided that downwind PM-10 particulate levels do not exceed 150 mcg/m<sup>3</sup> above the upwind level and provided that no visible dust is migrating from the work area.

2. If, after implementation of dust suppression techniques, downwind PM-10 particulate levels are greater than 150 mcg/m<sup>3</sup> above the upwind level, work must be stopped and a re-evaluation of activities initiated. Work can resume provided that dust suppression measures and other controls are successful in reducing the downwind PM-10 particulate concentration to within 150 mcg/m<sup>3</sup> of the upwind level and in preventing visible dust migration.

3. All readings must be recorded and be available for State (DEC and NYSDOH) and County Health personnel to review.

December 2009

#### Attachment D-2

NYSDEC TAGM #4031 Fugitive Dust Suppression and Particulate Monitoring Program at Inactive Hazardous Waste Sites

# TECHNICAL AND ADMINISTRATIVE GUIDANCE MEMORANDUM #4031

#### FUGITIVE DUST SUPPRESSION AND PARTICULATE MONITORING PROGRAM AT INACTIVE HAZARDOUS WASTE SITES

TO:	Regional Hazardous Waste Remediation Engrs., Bur. Directors & Section Chiefs
FROM:	Michael J. O'Toole, Jr., Director, Division of Hazardous Waste Remediation
SUBJECT:	DIVISION TECHNICAL AND ADMINISTRATIVE GUIDANCE MEMORANDUM FUGITIVE DUST SUPRESSION AND PARTICULATE MONITORING PROGRAM AT INACTIVE HAZARDOUS WASTE SITES
DATE:	Oct 27, 1989

Michael J. O'Toole, Jr. (signed)

#### 1. Introduction

Fugitive dust suppression, particulate monitoring, and subsequent action levels for such must be used and applied consistently during remedial activities at hazardous waste sites. This guidance provides a basis for developing and implementing a fugitive dust suppression and particulate monitoring program as an element of a hazardous waste site's health and safety program.

#### 2. Background

Fugitive dust is particulate matter--a generic term for a broad class of chemically and physically diverse substances that exist as discrete particles, liquid droplets or solids, over a wide range of sizes--which becomes airborne and contributes to air quality as a nuisance and threat to human health and the environment.

On July 1, 1987, the United States Environmental Protection Agency (USEPA) revised the ambient air quality standard for particulates so as to reflect direct impact on human health by setting the standard for particulate matter less than ten microns in diameter ( $PM_{10}$ ); this involves fugitive dust whether contaminated or not. Based upon an examination of air quality composition, respiratory tract deposition, and health effects,  $PM_{10}$  is considered conservative for the primary standard--that requisite to protect public health with an adequate margin of safety. The primary standards are 150 ug/m<sup>3</sup> over a 24-hour averaging time and 50 ug/m<sup>3</sup> over an annual averaging time. Both of these standards are to be averaged arithmetically.

There exists real-time monitoring equipment available to measure PM<sub>10</sub> and capable of integrating over a period of six seconds to ten hours. Combined with an adequate fugitive dust suppression program, such equipment will aid in preventing the off-site migration of contaminated soil. It will also protect both on-site personnel from exposure to high levels of dust and the public around the site from any exposure to any dust. While specifically intended for the protection of on-site personnel as well as the public, this program is not meant to replace long-term monitoring which may be required given the contaminants inherent to the site and its air quality.

#### 3. Guidance

A program for suppressing fugitive dust and monitoring particulate matter at hazardous waste sites can be developed without placing an undue burden on remedial activities while still being protective of health and environment. Since the responsibility for implementing this program ultimately will fall on the party performing the work, these procedures must be incorporated into appropriate work plans. The following fugitive dust suppression and particulate monitoring program will be employed at hazardous waste sites during construction and other activities which warrant its use:

- Reasonable fugitive dust suppression techniques must be employed during all site activities which may generate fugitive dust.
- Particulate monitoring must be employed during the handling of waste or contaminated soil or when activities on site may generate fugitive dust from exposed waste or contaminated soil. Such activities shall also include the excavation, grading, or placement of clean fill, and control measures therefore should be considered.
- Particulate monitoring must be performed using real-time particulate monitors and shall monitor particulate matter less than ten microns (PM<sub>10</sub>) with the following minimum performance standards:

Object to be measured: Dust, Mists, Aerosols Size range: <0.1 to 10 microns Sensitivity: 0.001 mg/m<sup>3</sup> Range: 0.001 to 10 mg/m<sup>3</sup> Overall Accuracy: ±10% as compared to gravimetric analysis of stearic acid or reference dust

Operating Conditions:

Temperature: 0 to 40°C Humidity: 10 to 99% Relative Humidity

Power: Battery operated with a minimum capacity of eight hours continuous operation

Automatic alarms are suggested.

Particulate levels will be monitored immediately downwind at the working site and integrated over a period not to exceed 15 minutes. Consequently, instrumentation

shall require necessary averaging hardware to accomplish this task; the P-5 Digital Dust Indicator as manufactured by MDA Scientific, Inc. or similar is appropriate.

- 4. In order to ensure the validity of the fugitive dust measurements performed, there must be appropriate Quality Assurance/Quality Control (QA/QC). It is the responsibility of the entity operating the equipment to adequately supplement QA/QC Plans to include the following critical features: periodic instrument calibration, operator training, daily instrument performance (span) checks, and a record keeping plan.
- 5. The action level will be established at 150 ug/m<sup>3</sup> over the integrated period not to exceed 15 minutes. While conservative, this short-term interval will provide a real-time assessment of on-site air quality to assure both health and safety. If particulate levels are detected in excess of 150 ug/m<sup>3</sup>, the upwind background level must be measured immediately using the same portable monitor. If the working site particulate measurement is greater than 100 ug/m<sup>3</sup> above the background level, additional dust suppression techniques must be implemented to reduce the generation of fugitive dust and corrective action taken to protect site personnel and reduce the potential for contaminant migration. Corrective measures may include increasing the level of personal protection for on-site personnel and implementing additional dust suppression techniques (see Paragraph 7). Should the action level of 150 ug/m<sup>3</sup> be exceeded, the Division of Air Resources must be notified in writing within five working days; the notification shall include a description of the control measures implemented to prevent further exceedences.
- 6. It must be recognized that the generation of dust from waste or contaminated soil that migrates off-site, has the potential for transporting contaminants off-site. There may be situations when dust is being generated and leaving the site and the monitoring equipment does not measure PM<sub>10</sub> at or above the action level. Since this situation

has the potential to migrate contaminants off-site, it is unacceptable. While it is not practical to quantify total suspended particulates on a real-time basis, it is appropriate to rely on visual observation. If dust is observed leaving the working site, additional dust suppression techniques must be employed. Activities that have a high dusting potential--such as solidification and treatment involving materials like kiln dust and lime--will require the need for special measures to be considered.

- The following techniques have been shown to be effective for the controlling of the generation and migration of dust during construction activities:
  - 1. Applying water on haul roads.
  - 2. Wetting equipment and excavation faces.
  - 3. Spraying water on buckets during excavation and dumping.
  - 4. Hauling materials in properly tarped or watertight containers.
  - 5. Restricting vehicle speeds to 10 mph.
  - 6. Covering excavated areas and material after excavation activity ceases.
  - 7. Reducing the excavation size and/or number of excavations.

Experience has shown that utilizing the above-mentioned dust suppression techniques, within reason as not to create excess water which would result in

unacceptable wet conditions, the chance of exceeding the 150 ug/m<sup>3</sup> action level at hazardous waste site remediations is remote. Using atomizing sprays will prevent overly wet conditions, conserve water, and provide an effective means of suppressing the fugitive dust.

8. If the dust suppression techniques being utilized at the site do not lower particulates to an acceptable level (that is, below 150 ug/m<sup>3</sup> and no visible dust), work must be suspended until appropriate corrective measures are approved to remedy the situation. Also, the evaluation of weather conditions will be necessary for proper fugitive dust control--when extreme wind conditions make dust control ineffective, as a last resort remedial actions may need to be suspended.

There may be situations that require fugitive dust suppression and particulate monitoring requirements with action levels more stringent than those provided above. Under some circumstances, the contaminant concentration and/or toxicity may require appropriate toxics monitoring to protect site personnel and the public. Additional integrated sampling and chemical analysis of the dust may also be in order. This must be evaluated when a health and safety plan is developed and when appropriate suppression and monitoring requirements are established for protection of health and the environment.

# Appendix E

Health and Safety Plan



Imagine the result

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# Appendix E Environmental Health and Safety Plan (E-HASP)

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

March 7, 2011

Christopher D. Keen Designated H&S Plan Writer

1. Phh

Charles P. Webster, CSP Designated H&S Plan Reviewer

Steves Teldman

Steven M. Feldman Project Manager

#### Appendix E Environmental Health and Safety Plan (E-HASP)

Former Dangman Park Manufactured Gas Plant Site Brooklyn, New York Site No. 224047 Index # A2-0552-0606

Prepared for: National Grid

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Our Ref.: B0036704.0001.00101

Date: March 7, 2011

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Former Dangman Park Manufactured Gas Plant Site

#### 1. Introduction

All work on this project will be carried out in compliance with ARCADIS' Health and Safety policies and procedures, and the Occupational Safety and Health Administration's Hazardous Waste Operations and Emergency Response regulation 29 CFR 1910.120. The design of this health and safety plan (HASP) conforms to the requirements of the ARC HSFS010 (HASP H&S Procedure). Specific health and safety information for the project is contained in this HASP. All personnel working on hazardous operations or in the area of hazardous operations shall read and be familiar with this HASP before doing any work. All project personnel shall sign the certification page acknowledging that they have read and understand this HASP.

Changes in the scope of the project or introduction of new hazards to the project shall require revision of the HASP by the HASP writer and reviewer, and approval by the Project Manager. The HASP Addendum Form and log table are included as Appendix A of this HASP.

## **ARCADIS**

## Appendix E Environmental Health and Safety Plan

Former Dangman Park Manufactured Gas Plant Site

#### 2. Project Site History and Requirements

On behalf of Brooklyn Union Gas d/b/a National Grid NY (National Grid), ARCADIS has prepared this HASP as a component of the Remedial Investigation (RI) Work Plan for the former Dangman Park Manufactured Gas Plant (MGP) site (Site) located at 486 Neptune Avenue, Brooklyn, New York.

The HASP should be used in conjunction with the RI Work Plan, the Field Sampling Plan (FSP), the Quality Assurance Project Plan (QAPP), and the Community Air Monitoring Plan (CAMP). The RI Work Plan presents the Site background and defines the field sampling program. The FSP describes the methods and procedures to be used for environmental sample collection during implementation of the RI field activities. The QAPP presents the quality assurance/quality control (QA/QC) procedures to be used during implementation of the RI Work Plan, as well as a description of the general field and laboratory procedures. The CAMP provides procedures to protect the downwind communities from potential airborne releases of constituents of concern during RI activities. The FSP, QAPP, and CAMP are provided as Appendices B, C, and D of the RI Work Plan, respectively.

#### 2.1 Site Background

Based on a review of available historical information, the Site was used as a MGP site from prior to 1895 until sometime between 1906 and 1930. The 1895 Sanborn map shows two gas holders, a retort house, two oil tanks, a tar tank, an engine room, a purifying house, and a shed. By 1906, the MGP Site was operated by the Brooklyn Borough Gas Company; an additional gas holder, generating house and cistern had been constructed, and the retort house and tar tank were no longer present. The MGP structures were removed sometime between 1906 and 1930. By 1930, the Site was occupied by a club house. By 1966, the Trump Village Shopping Center occupied the northern and central portions of the Site. Figure 2 of the RI Work Plan shows the approximate location of the former MGP structures.

#### 2.2 Site Description

Х	Active	Х	Secure		Industrial		Landfill	Service station
	Inactive		Unsecured	Х	Commercial		Well field	Water work
			Uncontrolled	Х	Residential		Railroad	Undeveloped
Other specify:								

#### Site Type: (Check as many as applicable)



Former Dangman Park Manufactured Gas Plant Site

The Site is located at 486 Neptune Avenue in the Borough of Brooklyn, New York City, Kings County, New York and occupies portions of two parcels that are identified by Tax Map Number: Block 7273, Lots 1 and 25. As shown on Figure 1 of the RI Work Plan, the Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is generally flat with an elevation of approximately 9 feet above mean sea level (msl). The closest natural surface water body is Coney Island Creek, which is located approximately 0.25 miles to the northwest of the Site.

The Site is located in the Coney Island community district of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5<sup>th</sup> Street to the east, a residential parcel to the south, and a commercial parcel to the west. The Site is currently occupied by a shopping center and a parking lot for a high-rise apartment building. The eastern portion of the shopping center is situated above the former MGP structures. As shown on Figure 2 of the RI Work Plan, the majority of the Site is either paved or developed with buildings.



Former Dangman Park Manufactured Gas Plant Site

Known Compounds	Source (soil/water/drum, etc.)		ncentration , mg/kg, mg/l)
		Lowest	Highest
Benzene	Soil	1.1 μg/kg	870 μg/kg
	Groundwater	2.1 μg/L	9,100 μg/L
Toluene	Soil	3.5 μg/kg	31,000 µg/kg
	Groundwater	1.4 μg/L	15,000 µg/L
Ethylbenzene	Soil	0.93 µg/kg	84,000 μg/kg
	Groundwater	9.6 µg/L	6,400 μg/L
Xylenes (Total)	Soil	0.71 μg/kg	51,000 μg/kg
	Groundwater	14 μg/L	3,200 μg/L
Total PAHs	Soil	0.034 mg/kg	1,219 mg/kg
	Groundwater	178 μg/L	7,986 μg/L
Cyanide	Soil	0.181 mg/kg	35.2 mg/kg
	Groundwater	5.5 μg/L	95.7 μg/L

The primary chemicals of concern (COCs) on this project are:

#### 2.3 List of Project Tasks and Scope of Work

 Task 1 – Drilling of Soil Borings, Temporary Monitoring Wells, and Monitoring Well Installation

Task 1 involves the following activities: ground penetrating radar (GPR) survey (i.e., surface geophysics), utility clearance, drilling soil borings/collecting soil cores for lithologic characterization and laboratory analysis, drilling temporary monitoring wells to collect groundwater samples for laboratory analysis, installation of monitoring wells, equipment decontamination, surveying of monitoring wells, and management of investigation-derived waste (IDW). The soil borings, temporary monitoring wells, and monitoring wells will be drilled using direct push, sonic, or hollow-stem auger (HSA) drilling techniques. It is planned that IDW (drill cuttings, purge water, development water, and decontamination water) will be containerized in Department of Transportation (DOT)-approved 55-gallon steel drums.



Former Dangman Park Manufactured Gas Plant Site

• Task 2 – Monitoring Well Sampling and Hydraulic Monitoring

Task 2 involves the sampling of monitoring wells to collect groundwater samples for laboratory analysis, the collection of water-level measurements, and management of IDW. The monitoring wells will be sampled using low-flow (minimal drawdown) groundwater sampling techniques. The water-level measurements will be collected using an electronic water-level indicator. It is planned that IDW (monitoring well purge water) will be containerized in DOT-approved 55-gallon steel drums.

• Task 3 – Temporary Soil Vapor Point Installation and Ambient Air Sampling

Task 3 involves the following activities: GPR survey, utility clearance, installation of temporary soil vapor points to collect soil vapor samples for laboratory analysis, collection of ambient air samples for laboratory analysis, and management of IDW. The temporary soil vapor points will be installed using soft dig techniques (i.e., hand excavation). It is planned that IDW (drill cuttings) will be containerized in DOT-approved 55-gallon steel drums.



Former Dangman Park Manufactured Gas Plant Site

#### 3. ARCADIS Organization and Responsibilities

#### 3.1 Project Manager/Task Manager

In planning and preparation of this project, the project manager and/or task manager has completed the project-specific H&S Stewardship Checklist & Project Hazard Analysis Worksheet. The project Hazard Analysis Worksheet was completed using the Hazard Analysis Risk Control (HARC) ranking process (ARCADIS H&S Procedure ARC HSMS002) (see Section 4 of this HASP). Additional responsibilities of the project manager and task manager are as follows:

- Review all applicable H&S Procedures, and ensure that project activities conform to all requirements.
- Obtain client-specific health and safety information and communicate with the client on health and safety issues.
- Communicate with the Site Safety Officer (SSO) on health and safety issues.
- Allocate resources for correction of identified unsafe work conditions.
- Ensure ARCADIS site workers have all training necessary for the project.
- Report all injuries, illnesses and near-misses to the Client H&S Resource (CHSR) or Project H&S Manager (PHSM), lead incident investigations, and ensure that any recommendations made are implemented.



Former Dangman Park Manufactured Gas Plant Site

#### 3.2 Other Project Team Responsibilities

Additional personnel designated to carry out H&S job functions for the project, and their responsibilities are listed below. The same person may fill more than one role:

ARCADIS Project Team	Responsibility and Tasks
On-Site Scientist, Technician, or	SSO
Engineer	Reviews and works in accordance with the components of this HASP.
	<ul> <li>Ensures that this HASP is available to and reviewed by all site personnel including subcontractors.</li> </ul>
	<ul> <li>Ensures that necessary site-specific training is performed (both initial and "tailgate" safety briefings.</li> </ul>
	<ul> <li>Ensures site visitors have been informed of the hazards related to ARCADIS work, and have signed the Site Visitors Log.</li> </ul>
	<ul> <li>Ensures that work is performed in a safe manner and has authority to stop work when necessary to protect workers and/or the public.</li> </ul>
	Coordinates activities during emergency situations.
	<ul> <li>Ensures that all necessary permits and safety information provided by the client is disseminated to other site personnel and is maintained in an organized manner.</li> </ul>
	<ul> <li>Communicates with the PM, CHSR and/or the PHSM on health and safety issues.</li> </ul>
	<ul> <li>Reports all injuries, illnesses and near-misses to the PM, CHSR and PHSM.</li> </ul>
	<ul> <li>Ensures that necessary safety equipment is maintained and used at the site.</li> </ul>
	<ul> <li>Contacts a health and safety professional for assistance in establishing the respiratory cartridge change schedule as required.</li> </ul>
On-Site Scientists, Technicians, and	Site Workers
Engineers	Reads and works in accordance with the components of this HASP.
	Reports all unsafe working conditions to the SSO.
	Reports all injuries, no matter how minor, to the SSO.
	Works in a safe manner.
	• Signs the HASP acceptance log in Appendix E of this HASP.



Former Dangman Park Manufactured Gas Plant Site

ARCADIS Project Team	Responsibility and Tasks					
Charles Webster	Project Health and Safety Manager (PHSM)					
	The PHSM oversees all aspects of the site safety program, and prepares site-specific health and safety guidance documents or addenda to this plan. The PHSM does not report to the Project Manager, and is separately accountable to the ARCADIS project team for site health and safety. The PHSM acts as the sole contact to regulatory agencies on matters of safety and health. Other responsibilities include:					
	<ul> <li>Overall authority for health and safety compliance and HASP conformance for the project.</li> </ul>					
	General health and safety program administration.					
	Conducts project health and safety audits as warranted.					
	Determines the level of personal protection required.					
	<ul> <li>Updates equipment or procedures based on information obtained during site operations.</li> </ul>					
	<ul> <li>Establishes air-monitoring parameters based on expected contaminants.</li> </ul>					
	Assists in injury, illness and near-miss investigations and follow-up.					
Charles Webster	Client Health and Safety Resource (CHSR)					
	The designated CHSR is responsible for :					
	Assisting the SSO in issues as they arise.					
	Performing site audits and assessments.					
	Assisting with near-miss/incident investigations.					
	<ul> <li>Serves as the liaison with corporate during H&amp;S regulatory issues as they may arise.</li> </ul>					

## **ARCADIS**

## Appendix E Environmental Health and Safety Plan

Former Dangman Park Manufactured Gas Plant Site

#### 4. Hazard Control

Effective hazard control begins with appropriate planning. ARCADIS has established several tools to help develop and implement hazard controls and avoid incidents at various risk levels. One of these tools, the Risk Assessment Matrix, is illustrated in Figure 1 below. H&S planners use the Risk Assessment Matrix (in conjunction with the Hazard Analysis Worksheet – Appendix B of this HASP) to rate hazards that could potentially be encountered on the job site. The results drive H&S planning for the entire project.

Risk Assess	sment Matrix	Likelihood Ratings**						
Consequence	ces Ratings*	A	В	С	D	E		
People Property		Never heard of in the world	Heard of incident in industry	Incident has occurred in ARCADIS Group	Happens several times a year in ARCADIS OpCo	Happens severa times a year at ARCADIS Worksite		
0 - No health effect	0 - No damage	Low	Low	Low	Low	Low		
1 - Slight health effect	1 - Slight damage	Low	Low	Low	Low	Low		
2 - Minor health effect	2 - Minor damage	Low	Low	Low	Medium	Medium		
3 - Major health effect	3 - Local damage	Low	Low	Medium	Medium	High		
4 - PTD or 1 fatality	4 - Major damage	Low	Medium	Medium	High	High		
5 - Multiple fatalities	5 - Extensive damage	Medium	Medium	High	High	High		

#### Figure 1. HARC - Risk Assessment Matrix (H&S Procedure ARC HSMS002)

The Hazard Analysis Worksheet is provided in Appendix B of this HASP.

The Risk Assessment Matrix can also be used in the field to evaluate and control risks as they arise. When practiced in the context of H&S Procedure ARC HSMS002, hazard control becomes an ongoing exercise that supports a safer working environment through continuous planning and preparation.

#### 4.1 Job Loss Analyses (JLAs), H&S Procedures and PPE

A JLA has been completed for each safety critical task, and are included in Appendix C of this HASP. Hazards identified on the Project Hazard Analysis Worksheet are addressed in the JLAs as well as control methods to protect employees and property from hazards. The JLA also lists the type of personal protective equipment (PPE) required for the completion of the project. A detailed list of PPE for the project is located in Appendix D of this HASP.



Former Dangman Park Manufactured Gas Plant Site

ARCADIS H&S Procedures applicable to this project are listed below. These procedures should be reviewed by the project manager, task manager and Site personnel. The CHSR should be contacted with any questions concerning the procedures.

The following health and safety management and general health and safety procedures apply to this project and are available on the ARCADIS Productivity EXchange (APEX):

- ARC HSMS002 Hazard Identification, Risk Assessment and Risk Control
- ARC HSMS010 Incident Reporting and Investigation
- ARC HSMS011 Root Cause Analysis and Solutions Development
- ARC HSGE001 Tailgate Health and Safety Meetings
- ARC HSGE004 First Aid/CPR
- ARC HSGE007 Hazard Communication
- ARC HSGE009 Stop Work Authority
- ARC HSGE008 Injury and Illness Prevention Program
- ARC HSGE010 Medical Monitoring Program
- ARC HSGE015 Personal Protective Equipment
- ARC HSGE024 Motor Vehicle Safety Program

The following specific procedures apply to this project and are located in Appendix G of this HASP:

- ARC HSFS012 Hazardous Waste Operations and Emergency Response
- ARC HSFS019 Utility Location Policy and Procedure
- ARC HSIH003 Benzene



Former Dangman Park Manufactured Gas Plant Site

• ARC HSIH008 – Hearing Conservation Health & Safety Procedure

#### 4.2 Field Health & Safety Handbook

The Field H&S Handbook is an ARCADIS document containing information about topic-specific health and safety requirements for the field. This handbook contains relevant general topics and is used as part of the overall HASP process. To aid in the consistency of the HASP process the handbook will be used as an informational source in conjunction with this HASP. Section III of the Field H&S Handbook is minimally required reading for this project.

The following handbook sections are additional required reading for this project:

- Section IV-E. Heavy Equipment
- Section IV-F. Hoisting and Rigging, Cranes and Derricks



Former Dangman Park Manufactured Gas Plant Site

#### 5. Hazard Communication (HazCom)

All project required chemicals must be handled in accordance with OSHA 29 CFR 1910.1200, ARCADIS-HazCom Procedure (ARC HSGE007), and the requirements outlined in the Field H&S Handbook. Table 1 lists all chemicals that will be brought, used, and/or stored on the Site by ARCADIS or its subcontractors. Material Safety Data Sheets (MSDS) for chemicals brought on site are included in Appendix F of this HASP. MSDS for other chemicals brought on site will be kept in the ARCADIS vehicle which transports the chemicals.

Table 1.	Master	Chemical	and Storage List
----------	--------	----------	------------------

Chemical Name	Estimated Quantity	Chemical Storage Location
Conductivity Standard Solution	500 mL	Sampling/Field Vehicle
YSI 3161 Conductivity Calibrator	1,000 mL	Sampling/Field Vehicle
ORP Calibration Solution	500 mL	Sampling/Field Vehicle
pH 4.00 Calibration Solution	500 mL	Sampling/Field Vehicle
pH 7.00 Calibration Solution	500 mL	Sampling/Field Vehicle
pH 10.00 Calibration Solution	500 mL	Sampling/Field Vehicle
Amco Clear: Turbidity Standard	500 mL	Sampling/Field Vehicle
Eye Saline Solution	500 mL	Sampling/Field Vehicle
ABC Fire Extinguisher Powder	10 lbs	Sampling/Field Vehicle
Gasoline, All Grades	150 L	Sampling/Field Vehicle
Motor Oil	1 Qt	Sampling/Field Vehicle
Hydrochloric Acid	<100 mL	Sampling/Field Vehicle
Sodium Hydroxide	<500 mL	Sampling/Field Vehicle
Micro-90 Cleaning Solution	1 Qt	Sampling/Field Vehicle
Isobutylene Gas	17 L	Sampling/Field Vehicle
BioSolve Hydrocarbon Mitigation Agent	5 gal	Sampling/Field Vehicle



Former Dangman Park Manufactured Gas Plant Site

#### 5.1 Chemical Hazards

Air monitoring will be conducted as outlined in this HASP to collect exposure data for chemicals of concern (COCs) or for chemicals brought on site for use. Table 2 lists the properties of chemicals that will be encountered at the Site.

#### Table 2. Chemical Hazard Information

Chemical Name	IP (eV)	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hr TWA <sup>1</sup> (ppm)	IDLH (NIOSH) (ppm)	STEL (ppm)	Source TLV/PEL
Benzene	9.24	34-119	Routes of Entry: Inhalation,	0.5	500	2.5	TLV
			Absorption, Ingestion,				
			Contact				
			Exposure Symptoms:				
			Irritation eyes, skin, nose,				
			respiratory system;				
			dizziness; headache, nausea,				
			staggered gait; anorexia,				
			lassitude (weakness,				
			exhaustion); dermatitis; bone				
			marrow depression;				
			[potential occupational				
			carcinogen]				
Benzo[a]pyrene			Routes of Entry: Inhalation,	0.2	80	NA	(as
(Coal tar pitch volatiles)			Ingestion, Contact	mg/m <sup>3</sup>	mg/m <sup>3</sup>		benzene
			Exposure Symptoms:				soluble
			Eye and skin irritation,				aerosol)
			respiratory tract irritation.				ACGIH
			Potential cancer causing				
			chemical				
Cyanides: calcium, potassium,	NA	NA	Routes of Entry: Inhalation,	5	25	NA	TLV
and sodium			Absorption, Ingestion,	mg/m <sup>3</sup>	mg/m <sup>3</sup>		
			Contact	(skin)			
			Exposure Symptoms:				
			Asphyxiation and death can				
			occur; weakness, headache, and				
			confusion; nausea and vomiting;				
			increased respiratory rate; slow				
			respiratory gasping; irritated eyes and skin				
1							



Former Dangman Park Manufactured Gas Plant Site

Chemical Name	IP (eV)	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hr TWA <sup>1</sup> (ppm)	IDLH (NIOSH) (ppm)	STEL (ppm)	Source TLV/PEL
Fluorene			Routes of Entry: Inhalation, Contact Exposure Symptoms: It is irritating to the skin, eyes, and respiratory tract.	NE			
Ethylbenzene	8.76	0.09-0.6	Routes of Entry: Inhalation, Ingestion, Contact Exposure Symptoms: Irritation eyes, skin, mucous membrane; headache; dermatitis; narcosis, coma	100	800	125	TLV
Naphthalene	8.12	0.0095- 0.64	Routes of Entry: Inhalation, Absorption, Ingestion, Contact Exposure Symptoms: Irritation eyes; headache, confusion, excitement, malaise (vague feeling of discomfort); nausea, vomiting, abdominal pain; irritation bladder; profuse sweating; jaundice; hematuria (blood in the urine), renal shutdown; dermatitis, optical neuritis, corneal damage	10	250	15	TLV



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Chemical Name	IP (eV)	Odor Threshold (ppm)	Routes of Entry/ Exposure Symptoms	8-hr TWA <sup>1</sup> (ppm)	IDLH (NIOSH) (ppm)	STEL (ppm)	Source TLV/PEL
Toluene	8.82	0.16-37	Routes of Entry: Inhalation, Absorption, Ingestion, Contact Exposure Symptoms: Irritation eyes, nose; lassitude (weakness, exhaustion), confusion, euphoria, dizziness, headache; dilated pupils, lacrimation (discharge of tears); anxiety, muscle fatigue, insomnia; paresthesia; dermatitis; liver, kidney damage	50	500	NA	TLV
Xylene (o-, m-, and p- isomers)	8.44- 8.56	0.08-40	Routes of Entry: Inhalation, Absorption, Ingestion, Contact Exposure Symptoms: Irritation eyes, skin, nose, throat; dizziness, excitement, drowsiness, incoordination, staggering gait; corneal vacuolization; anorexia, nausea, vomiting, abdominal pain; dermatitis	100	900	150	TLV

<sup>1</sup>The TLV (Threshold Limit Value) from the American Conference of Governmental Industrial Hygienists (ACGIH) is listed unless the PEL (Permissible Exposure Limit), designated by OSHA, is lower.

See Section 7 of this HASP for information on air monitoring requirements.



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#### 6. Tailgate Meetings

Tailgate safety briefings will be conducted at least twice daily, once at the beginning of the work day, again after lunch, or as tasks/hazards change. Each tailgate safety briefing will be documented on the form included in Appendix E of this HASP.



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#### 7. Personal Exposure Monitoring and Respiratory Protection

#### 7.1 Air Monitoring

Air monitoring will be conducted to evaluate airborne levels of COCs. The monitoring results will dictate work procedures and the selection of PPE for ARCADIS employees and ARCADIS visitors only. The monitoring devices to be used, at a minimum, are an MIE personal DataRAM pDR-1000 particulate monitor (or equivalent) and a combination lower explosive limit (LEL)/oxygen (O<sub>2</sub>)/hydrogen sulfide (H<sub>2</sub>S)/carbon monoxide (CO)/photoionization detector (PID) with a 10.6 eV lamp. The RAE Systems MultiRAE Plus is an example of this type of instrument. Colorimetric detector tubes for benzene may be needed if organic vapor action levels are met.

Monitoring for organic vapors and particulates will be conducted in the exclusion zone during all ground intrusive activities. Monitoring data will be recorded on the Real Time Exposure Monitoring Data Form (Appendix E of this HASP).

If ARCADIS and one or more ARCADIS subcontractors are working in an area, one subcontractor may conduct direct-reading air monitoring and share the results with the other subcontractors working in the area. In this situation, subcontractors should coordinate air monitoring through a mutually-agreed upon air monitor. The ARCADIS PHSM will be responsible for utilizing the air monitoring results to specify the appropriate health and safety precautions for ARCADIS personnel only.

Air monitoring should be conducted continuously with the LEL/O<sub>2</sub>/H<sub>2</sub>S/CO meter during work activities in areas where flammable vapors or gases are suspected. Work activity must stop where tests indicate the concentration of flammable vapors exceeds 10% of the LEL at any location. Such an area should be ventilated to reduce the concentration to an acceptable level. Continuous carbon monoxide monitoring is required to be conducted if internal combustion engines (drill rig, etc.) are used. During operations that may cause airborne particulates (e.g., drilling and well installation), a particulate monitor should be used to measure airborne concentrations of total particulate material. At a minimum, readings should be measured and recorded on an hourly basis on an air monitoring log (Appendix E of this HASP) or in a field notebook.

#### 7.2 Noise Monitoring

Noise monitoring may be conducted as required with a calibrated Sound Level Meter capable of accurately measuring noise levels. Hearing protection is mandatory for all



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employees in noise hazardous areas, such as around heavy equipment. As a general rule, sound levels that cause speech interference at normal conversation distance should require the use of hearing protection.

#### 7.3 Monitoring Equipment Maintenance and Calibration

All direct-reading instrumentation calibrations should be conducted under the approximate environmental conditions the instrument will be used. Instruments must be calibrated before use, noting the reading(s) and any adjustments that are necessary. All air monitoring equipment calibrations, including the standard used for calibration, must be documented on a calibration log or in the field notebook. All completed health and safety documentation/forms must be reviewed by the PHSM and maintained by the SSO.

All air monitoring equipment will be maintained and calibrated in accordance with the specific manufacturer's procedures. Preventive maintenance and repairs will be conducted in accordance with the respective manufacturer's procedures. When applicable, only manufacturer-trained and/or authorized personnel will be allowed to perform instrument repairs or preventive maintenance.

If an instrument is found to be inoperative or suspected of giving erroneous readings, the PHSM must be responsible for immediately removing the instrument from service and obtaining a replacement unit. If the instrument is essential for safe operation during a specific activity, that activity must cease until an appropriate replacement unit is obtained. The PHSM will be responsible for ensuring a replacement unit is obtained and/or repairs are initiated on the defective equipment.

#### 7.4 Action Levels

Table 3 presents airborne contaminant action levels that will be used to determine the procedures and protective equipment necessary based on conditions as measured at the Site.



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#### Table 3. Airborne Contaminant Action Levels

Parameter	Reading	Action
Total Organic Vapors (measured with a PID) <sup>1</sup>	0 ppm to <u>&lt;</u> 0.5 ppm	Normal operations; continue hourly breathing zone monitoring.
	> 0.5 ppm to < 5 ppm	Increase monitoring frequency to every 15 minutes. Use secondary monitoring device to screen for the presence of benzene (see below).
	<u>&gt;</u> 5 ppm to <u>&lt;</u> 10 ppm	Upgrade to Level C PPE; continue screening for benzene.
	> 10 ppm	Stop work; investigate cause of reading.
Benzene (from colorimetric tube)	≥ 0.5 ppm to 10 ppm	Upgrade to Level C PPE.
	> 10 ppm	Stop work; investigate cause of reading.
Total Particulates (measured with a personal DataRAM) <sup>2</sup>	0 to 0.100 mg/m <sup>3</sup> above background	Normal operations.
	> 0.100 mg/m <sup>3</sup> above background	Initiate wetting of work area to control dust, upgrade to Level C if dust control measures do not control dust within 15 minutes. Monitor downwind impacts.
	> 0.150 mg/m <sup>3</sup> in breathing zone or at downwind perimeter of work area	Stop work, investigate cause of reading, and ventilate area.
Oxygen	<u>&lt;</u> 19.5%	Stop work, evacuate work area, investigate cause of reading, and ventilate area.
	> 19.5% to < 23.5%	Normal operations.
	<u>&gt;</u> 23.5%	Stop work, evacuate work area, investigate cause of reading, and ventilate area.
Carbon Monoxide	0 ppm to <u>&lt;</u> 20 ppm	Normal operations.
	> 20 ppm	Stop work, evacuate work area, investigate cause of reading, and ventilate area.
Hydrogen Sulfide	0 ppm to <u>&lt;</u> 5 ppm	Normal operations.
	> 5 ppm	Stop work, evacuate work area, investigate cause of reading, and ventilate area.
Flammable Vapors (LEL)	< 10% LEL	Normal operations.
	<u>&gt;</u> 10% LEL	Stop work, ventilate area, investigate source of vapors.



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#### Notes:

- 1) Readings for Total Organic Vapors are at breathing zone height, measured with a calibrated PID.
- 2) Readings for particulates are for results measured with a calibrated personal DataRAM. Dust sampling instruments provide "total dust" levels, and do not differentiate between contaminated and non-contaminated dust particulates. Dust action levels are based upon total dust and not respirable dust levels.

Note – Use of respiratory protection is not anticipated or planned. If exposure monitoring readings in the **breathing zone** are above action levels, work is to stop and the PM and CHSR informed. Engineering controls such as modifying work and ventilation may be considered. PM and CHSR must concur before respirators will be utilized and respirators will be considered a last resort.



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#### 8. Medical Surveillance

Medical surveillance requirements for the project are provided on the Project Manager/Task Manager H&S Stewardship Checklist & Project Hazard Analysis Worksheet (Appendix B of this HASP). All medical surveillance requirements as indicated on the worksheet must be completed and Site personnel medically cleared before being permitted on the project Site.



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#### 9. General Site Access and Control

The SSO will coordinate access and control security at the work Site. As the work dictates, the SSO will establish a work area perimeter. The size of the perimeter will be based on the daily task activities and will be discussed with all project personnel during the tailgate meeting and then documented on the tailgate meeting form. Control zones will be demarcated by either visual or physical devices and will be monitored for effectiveness by the SSO.

Only authorized personnel will be allowed beyond the perimeter. Other Site workers and visitors to the Site should be kept out of the work Site. If visitors need access to the Site, the SSO will escort the visitor at all times. All visitors will log in and out with the SSO. The visitor log sheet is included in Appendix E of this HASP.



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#### 10. Decontamination Control Zones and Procedures

Part of required reading for this HASP includes reviewing the Field H&S Handbook, Section III-G Site Security, Work Zones and Decontamination for HAZWOPER site zones. The decontamination procedures outlined in the Field H&S Handbook are provided for typical Level D and Level C ensembles.

The zones will be designated by traffic cones, barricades, signs, caution tape, or other means effective in identifying the different areas. The SSO will establish control boundaries for the exclusion zone, contamination reduction zone, and the support zone. The zones will be identified by the SSO during tailgate meetings and documented on the meeting form. Entrance and exit to the exclusion zone will only be through controlled access points established for each work area.



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#### 11. Emergency Action Plan (EAP)

In the event that an injury, over-exposure or spill has occurred, an EAP will be implemented. Appendix H of this HASP provides the EAP and notifications for the project. All employees working on this project must be shown the location and proper use of all emergency equipment prior to beginning work on the project.



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## 12. Department of Transportation (DOT) Dangerous Goods Shipping Requirements

ARCADIS has policies in place for transporting small quantities of hazardous materials and for offering for shipping via ground or air. These policies are designed to meet the applicable requirements. As such, only ARCADIS staff that have been trained in the proper methods to prepare and ship hazardous materials are authorized to do so. Tasks associated with the packaging, labeling, marking, and preparation of hazardous materials for shipping or transport must have all appropriate and applicable training.

#### 12.1 Materials of Trade (MOT)

DOT allows for a small amount of hazardous materials that are used in or an inherent part of our work to be transported in company vehicles. This includes things like gasoline, paint, small compressed gas cylinders, calibration gas, etc. To transport these:

- Staff will complete Materials of Trade training.
- Vehicles used in transportation to and from off-site work locations will be in conformance with ARCADIS vehicle safety procedures.

Hazardous materials will be transported as described above as a result of the activities covered in this HASP. Site personnel who transport materials mentioned above will complete the Hazardous Materials Transportation Form included in Appendix E of this HASP.

#### 12.2 Department of Transportation

Staff who collect, prepare, package, mark, label, complete shipping declarations, offer shipments to a transporter, directly transport or are engaged in other activities associated with the transportation of Hazardous Materials (referred to as Dangerous Goods in Canada and by the International Air Transport Association [IATA]) will have appropriate and applicable training. DOT requires all individuals who participate in hazmat shipping including activities such as completing the paperwork (but not signing it), filling a container with a hazardous material (including filling a drum with drill cuttings or purge water), marking, labeling, and packaging the hazardous material, etc., have awareness level training on the DOT requirements. DOT requires additional job function training for those who conduct specific activities including:



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- Staff who have to sign shipping papers or manifests, are listed as the 24-hour emergency contacts on shipping and have the responsibility for identifying, classifying, packaging, marking, and labeling HazMat packages, and/or are directing or overseeing others who do these tasks will become certified through the completion of additional training.
- The above training allows the offering employee to ship only by ground. If the shipment is to be offered for air transport, additional training is required.

Shipments as described above will be made as a result of the activities covered in this HASP. Site personnel shipping hazardous materials will complete the Hazardous Materials Shipment Form included in Appendix E of this HASP.



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## 13. Loss Prevention System<sup>™</sup> (LPS<sup>™</sup>) and Loss Prevention Observations (LPOs)

As part of any project, no matter how simple or complex, LPOs should be conducted when practical and when able to integrate into normal business activities. LPOs should be scheduled based on the risk of the tasks being performed, and should be conducted for different tasks and at different times. Completion of LPOs should be documented on the tailgate meeting form.

Identified Task for LPO	Schedule Date	Observer Name	Observee Name	Feedback Supervisor Name
Drilling	TBD	TBD	TBD	TBD
Groundwater	TBD	TBD	TBD	TBD
Sampling				

The following table outlines the LPO plan for the project:

## **ARCADIS**

### Appendix E Environmental Health and Safety Plan

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#### 14. Subcontractors

A copy of this HASP is to be provided to all subcontractors prior to the start of work so that the subcontractor is informed of the hazards at the Site. While the ARCADIS HASP will be the minimum health and safety requirements for the work completed by ARCADIS and its subcontractors, each subcontractor, in coordination with ARCADIS health and safety personnel, is expected to perform its operations in accordance with its own HASP, policies and procedures unique to the subcontractor's work to ensure that hazards associated with the performance of the work activities are properly controlled. Copies of any required safety documentation for a subcontractor's work activities.

In the event that the subcontractor's procedures/requirements conflict with requirements specified in this HASP, the more stringent guidance will be adopted after discussion and agreement between the subcontractor and ARCADIS project health and safety personnel. Hazards not listed in this HASP, but known to the subcontractor or known to be associated with the subcontractor's services, must be identified and addressed to the ARCADIS project or task manager and SSO prior to beginning work operations.

If the subcontractor prefers to adopt this HASP, the <u>"Subcontractor</u> <u>Acknowledgement Memo" must be signed and dated by the subcontractor's</u> <u>management and placed in the project file.</u> Once the signed memo is received by the project manager, an electronic version of our HASP can be submitted to the subcontractor to use as their own. Subcontractors working at the Site will need to have this plan with them, and will also need to sign the Subcontractor HASP receipt signature page of the ARCADIS HASP (Appendix E of this HASP). Subcontractors are responsible for the H&S of their employees at all times, and have the authority to halt work if unsafe conditions arise.

The Project/Task Manager and SSO (or authorized representative) has the authority to halt the subcontractor's operations and to remove the subcontractor or subcontractor's employee(s) from the Site for failure to comply with established health and safety procedures or for operating in an unsafe manner.



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## 15. Project Personnel HASP Certification

All Site project personnel will sign the certification signature page provided in Appendix E of this HASP.

# **ARCADIS**

## Appendix A

HASP Addendum Pages and Log Table



## **Addendum Page**

This form should be completed for new tasks associated with the project. The project manager and/or task manager should revise the Project Hazard Analysis Worksheet with the new task information and attach to this addendum sheet. JLAs should be developed for any new tasks and attached as well.

Review the addendum with all site staff, including subcontractors, during the daily tailgate briefing, and complete the tailgate briefing form as required. Attach a copy of the addendum to all copies of the HASP including the site copy, and log in the Addendum Log Table A-1 on the next page.

Addendum Number:	Project Number:	
Date of Changed Conditions:	Date of Addendum:	

Description of Change that Results in Modifications to HASP:

Signed:

Signed:

Site Safety Officer

Signed:

H&S Plan Writer

Project Manager

Signed:

H&S Plan Reviewer



## Addendum Log Table

Addendums are to be added to every copy of the HASP, and logged on Table A-1 to verify that all copies of the HASP are current:

Table A-1 Addendum Log Table

Addendum Number	Date of Addendum	Reason for Addendum	Person Completing Addendum
1			
2			
3			
4			
5			
6			
7			
8			
9			
10			

# **ARCADIS**

Appendix B

Project Hazard Analysis Worksheet

Inint: National Grid Principal-In Charge: James Nuss roject / Task Manage: Steven Feldman By: Christopher Keen Date: 9/29/10 ARCADIS Project Hazard Analysis Worksheet TRACK Recognize and Assess the Hazards for the Project and all of the tasks and assess them using High (H), fedum (M), Low (L). Use the drop down list in each "Assess" and the entire project and all of the tasks and assess them using High (H), fedum (M), Low (L). Use the drop down list in each "Assess" and the entire project and all of the tasks and assess them using High (H), fedum (M), Low (L). Use the drop down list in each "Assess" and the entire project and all of the tasks and assess them using High (H), fedum (M), Low (L). Use the drop down list in each "Assess" and the entire project and all of the tasks and assess them using High (H), fedum (M), Low (L). Use the drop down list in each "Assess" feecognize the Hazards Head to the tasks and assess the Hazards Below Heads Low Uniting Radiation Huzards Below Heads Low Uniting Radiation Noise Use Mathematical Hazard, Low Uniting Radiation Noise Use Mathematical Hazard, Low Uniting Radiation Noise Use Mathematical Hazard, Low Use Mathematical Low Use Control the Hazard, LOW Noise Use Noise Waither Low Use Noise Waither Low Noise Use Noise Waither Control the Actor Second Low Dote: Noise Use Noise Waither Mathematical Low Use Noise Waither Low Noise Use Noise Use Noise Waither Mathematical Hazards will be controled) Dote: Noise Use Noise Wile be provided will be like breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during Waither Pres Souther Corroling Radiation, Administrative and engineering controls will also de (e.g., enployees will keep Nytated and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during Waither Mathematical Hazards: Note: Mathematical Hazards will be controlled) Dometer Hazard		Pr	oject Haza	rd Analysis Page	1		
Project / Task Manager Seven Feldman Seven Feldman Seven Feldman Seven Feldman Seven Feldman Seven Feldman Seven ARCADIS Project Hazard Analysis Worksheet TRACK  ARCADIS Project Hazard Analysis Worksheet TRACK  ARCADIS Project Instance	roject Name:	Former Dangman Park MGP Site		Project Number:	B0036704	B0036704.0001	
Project / Task Manager:         Seven Feldman         By:         Christopher Ken         Date:         923/0           ARCADIS Project Hazard Analysis Worksheet TRACK           Compute and Assess the Hazards for the Project or each potentia hazards.           Recipite the Head Assess the Hazards for the Project or each potentia hazards.           Image: Second	lient:	National Grid		Principal-In Charge:	James Nu	ISS	
TRACK           Concentration of the function for the number of action of the function for the number project and all of the tasks and assess them using High (H), each most case conditions for the number project and all of the tasks and assess them using High (H), each most case conditions for the number project and all of the tasks and assess them using High (H), each most case conditions for the number project and all of the tasks and assess them using High (H).           Image: task of the task of the task of the tasks and assess them using High (H).         Image: task of the task of tasks of the task of the tasks and assess them using High (H).           Image: task of task of the task of tasks of tas	roject / Task Manager:		•	Christopher Keen	Date:	9/29/10	
Percential Hazards the Hazards for the Project or each potential hazard, determine the worst case conditions for the entire project and all of the tasks and assess them using High (H), each musing		ARCADIS	Project Ha	zard Analysis Work	sheet		
or each proteinial hazard, elemine the worst case conditions for the entire project and all of the tasks and assess the using high (th), eledium (th), Low (L). Use the drap down list in each "Assess" loci. If a hazards on the site, leaves the "Assess" bot blank.           Image: transmission of the second seco			T	RACK			
Hards         Or         Harards         Hear           Hear         Low         Horizing Radiation         Heards         Heards           Noise         Low         Horizing Radiation         Heards         Heards           Nysical Hazards:         WalkingWorking surfaces (ficulde alphriphil & Roor/wal yrable Dust         Low         Non-horizing Radiation         Generator           Vable Dust         Low         Poor lighting         Low         Generator           Other:         Operational Heards         Low         Generator           Other:         Operational Heards         Low         Generator           Other:         Operation Heards         Low         Generator           Operation proteine regimeering controls will be controlled)         Dow         Generator           Sprotechures will be reviewed prot to beginning work and will be followed during work. In addition, administrative and engineering controls will also de (e.g., employees will keep hydrated and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during will safety briefings.           Hermical Hazards:         Flammable/ Combustible         Corrosive         Low         List the Names of the Mage Chemicals Below           Control the Hazards:         Hear mathle/ Conducter         Low         Corrosive         Low         Explosive <td>or each potential hazard, de</td> <td>etermine the worst case cor</td> <td>ditions for the</td> <td></td> <td></td> <td></td>	or each potential hazard, de	etermine the worst case cor	ditions for the				
Heat         Low         Hoise/Pis           Noise         Low         Non-inning Radiation			Assess	Recognize the Hazards	Assess	List Types of other Physical	
Cold         Low         Ionizing Relation         Image: Construct Relation           hysical Hazards:         Service (include sight)         Low         Generator           Visible Cust         Low         Electricity         Low         Generator           Visible Cust         Low         Electricity         Low         Generator           Other:         Overhead Hazards         Low         Generator           Other:         Nore: Mark With an 3'C         Low         Generator           Control the Hazard:         (Briefly describe how the identified hazards will be controlled)         Generator         Generator           Spropetares will keep hydrated and take breaks as needed to avoid heat extausion) and awareness of site hazards will be discussed during with stable for each hask. ARCA         Approx           As Sproedures will keep hydrated and take breaks as needed to avoid heat extausion) and awareness of site hazards will be discussed during with stable for each hask. ARCA         Approx           Control the Hazards:         Flammable/         Corrosive         Low         List the Names of the Maging will keep hydrated and take breaks as needed to avoid heat extausion) and awareness of site hazards will be discussed during with stable for each hask.           Presenting         Low         Corrosive         Low         Entythenzene, Righ? Fluorene, Righ?           Control the Hazards:         F			Low	Holes/Pits		Hazarus Below	
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signifying all site floor/wall         Medium         Low         Control           Visible Dust         Low         Poor lighting         Low		Walking/Working		Electricity			
Visible Dust         Low         Poor (righting Other:         Low           Other:         Overhead Hazards         Low           Other:         Overhead Hazards         Low           Other:         None: Mark with an 'X'         Image: Control the Hazard's (Briefly describe how the identified hazard's will be controlled)           proportiate personal protective equipment (PPE) as outlined in the Job Loss Anapsis (LA) will be used by all on-site employees for each task. ARCA So procedures will be enviced prior to beginning work and will be followed during work. In addition, administrative and engineering controls will allow Granticals Below           Beinzene, B(a)P. Naphthalene Toxic         Low         Corrosive         Low         List the Names of the Mage Chemicals Below           Compressed gas         Toxic         Low         Explosive         Difference, B(a)P. Naphthalene Toulene, Xylene           Organic peroxide         Irritant         Low         Enzone, B(a)P. Fluorene, Ethylbenzene, Naphthalene, Toulene, Xylene           Oxidizer         Vater reactive         Carrinogen         Low         Benzene, B(a)P. Fluorene, Ethylbenzene, Naphthalene, Toulene, Xylene           Outrol the Hazard:         Briefly describe how the identified hazard's will be controlled)         Toulene, Xylene           Organic peroxide         Irritant         Low         Benzene, B(a)P. Fluorene, Ethylbenzene, Naphthalene, Toulene, Xylene           Outrot the H	Physical Hazards:	slip/trip/fall & floor/wall	Medium		Low	Generator	
LASER         Severa Weather         Low           Ordersed Hazards         Low         Control         Low           Ontrol the Hazard: (Briefly describe how the identified hazards will be controlled)         propriate presonant protective equipment (PPE) as outlined in the Juck Loss Anapsit, LUA will be used by all on-site employees for each task. ARCA SC procedures will be provide and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during work. In addition, administrative and engineering controls will also add (a.g. employees will keep hydrated and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during vork. In addition, administrative and engineering controls will also add (a.g. employees will keep hydrated and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during vork. In addition, administrative and engineering controls will also add (a.g. employees will keep hydrated and take breaks as needed to avoid heat exhaustion) and awareness of site hazards will be discussed during vork. In addition, administrative and engineering controls will also addition will be related to avoid heat exhaustion of the Majg Compressed gas           Flammable/         Low         Corrosive         Low         List the Names of the Majg Compressed gas           Ordizer         Granic periods         Intrant         Deep temployee for periods         Deep temployee for periods           Outered         Organic periods         Intrant         Deep temployee for periods         Dev temployee for periods           Outered         Sensitizer			Low	Poor lighting	Low		
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Control the Hazard: (Birligh describe how the identified hazards will be controlled)           propriate presonal protective equipment (PPE) as outlined in the Job Loss Anaysis (JLA) will be used by all on-site employees for each task. ARCA Sproedures will be reviewed prior to beginning work and will be followed during work. In addition, administrative and engineering controls will also solutions of the Majo Corrosive           Low         List the Names of the Majo Corrosive         Low         List the Names of the Majo Chemicals Below           Bernzene, B(a)P, Naphthalene         Corrosive         Low         List the Names of the Majo Chemicals Below           Compressed gas         Toxic         Low         Bernzene, B(a)P, Naphthalene           Organic periodic         Irritant         Bernzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene           Oxidizer         Sensitizer         Toluene, Xylene           Oxidizer         Sensitizer         Toluene, Xylene           Oxidizer         Bernzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene         Distrumes/           Distremes/         Low         None: Mark with an "X"         Distrumes/           Printiable reactive         Low         None: Mark with an "X"         Distrumes/           Opropriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during illy safety briefings before starting work to create awareness to chemical haza		Other:		Overhead Hazards	Low		
propriate personal protective equipment (PPE) as outlined in the Job Loss Analysis (JLA) will be used by all on-site employees for each task. ARCA Sprocdures will be reviewed prior to beginning work and will be followed during work. In addition, administrative and engineering controls will also Sprocdures will be reviewed prior to beginning work and will be followed during work. In addition, administrative and engineering controls will also Sprocdures will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards.         hemical Hazards:       Flammable/ Combustible       Corrosive       Low       List the Names of the Magnetic Sprocess of the sproces of the sprocess of the sprocess of the sproce		Other:		None: Mark with an "X"			
propriate personal protective equipment (PPE) as outlined in the Job Loss Analysis (JLA) will be used by all on-site employees for each task. ARCA Sprocdures will be reviewed prior to beginning work and will be followed during work. In addition, administrative and engineering controls will also Sprocdures will be reviewed prior to beginning work and will be followed during work. In addition, administrative and engineering controls will also Sprocdures will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards will be discussed during the sprocess of site hazards.         hemical Hazards:       Flammable/ Combustible       Corrosive       Low       List the Names of the Magnetic Sprocess of the sproces of the sprocess of the sprocess of the sproce	ontrol the Hazard (D	riofly describe how the idea	tified bezorda	will be controlled)			
hemical Hazards:       Compressed gas       Toxic       Low       Benzene, B(a)P, Naphthalene, Toluene, Xylene         hemical Hazards:       Organic peroxide       Initiant       Benzene, B(a)P, Fluorene, Low       Ethylbenzene, Naphthalene, Toluene, Xylene         Oxidizer       Sensitizer       Low       Ethylbenzene, Naphthalene, Toluene, Xylene         Oxidizer       Sensitizer       Mutagen       Ethylbenzene, Naphthalene, Toluene, Xylene         DustFumes/       Low       Mutagen       Benzene, B(a)P         DustFumes/       Low       None: Mark with an "X"       Benzene, B(a)P         Outrol the Hazard: (Briefly describe how the identified hazards will be controlled)       propriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ily safety briefings before starting work to create awareness to chemical hazards.         None: Mark with an "X"       Docks - marine       Scaffolding       Environmental / Equipment Hazards Below         nvironmental/       Trenching/excavation       Ladders       Low       Calibration gases         Doiks - marine       Operations       Medium       Gas calinding       Diving operations         Doiks - marine       Operations work       Ralinad work       Medium       Medium         Water operations work       Ralinad work       Energize			Low	Corrosive	Low	List the Names of the Majo Chemicals Below	
hemical Hazards:       Explosive       Highly toxic       Description         Organic peroxide       Irritant       Low       Benzene, B(a)P, Fluorene, Raphthalene, Toluene, Xylene         Oxidizer       Sensitizer       Irritant       Benzene, B(a)P, Fluorene, Raphthalene, Toluene, Xylene         Oxidizer       Carcinogen       Low       Benzene, B(a)P         Unstable reactive       Mutagen       Benzene, B(a)P         Dust/Furmes/       Low       None: Mark with an "X"       Particulates         Particulates       Low       None: Mark with an "X"       Particulates         Control the Hazard: (Briefly describe how the identified hazards will be controlled)       propriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.         nvironmental/       Trenching/excavation       Ladders       Environmental / Equipmer Hazards         quipment Hazards:       Forklifts       Manifts       Diving operations       Calibration gases         Diving operations       Welding       Medium       Gas divinders       Low       Calibration gases         Diving operations       Welding       Calibration gases       Diving operations       Medium       Medium         Overhead/ Undergr				Toxic	Low	Benzene, B(a)P, Naphthalene,	
hemical Hazards:       Low       Ethylbenzene, Naphthalene, Toluene, Xylene         Oxidizer       Sensitizer       Image: Carcinogen       Low         Water reactive       Mutagen       Image: Carcinogen       Low         DustFumes/ Particulates       Low       None: Mark with an "X"       Image: Carcinogen         Ontrol the Hazard:       (Briefly describe how the identified hazards will be controlled)         propriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during aily safety briefings before starting work to create awareness to chemical hazards.         Image: Carcinogen construction activities       Medium       Cranes/Hoists/Rigging       List Types of Other Environmental / Equipmer Hazards.         Image: Construction activities       Manifits       Image: Carcinogen       Heavy machinery         Image: Construction activities       Manifits       Image: Carcinogen       Hazards Below         Image: Construction activities       Manifits       Image: Carcinogen       Image: Carcinogen         Image: Construction activities       Manifits       Image: Carcinogen       Image: Carcinogen         Image: Construction activities       Manifits       Image: Carcinogen       Image: Carcinogen         Image: Construction activities       Roadway work       Medium       Image: Carcinogen						Toluene, Xvlene	
Oxidizer         Sensitizer         Low           Water reactive         Carcinogen         Low           Dust/Furnes/ Particulates         Low         None: Mark with an *X*           Optor1 the Hazard:         (Briefly describe how the identified hazards will be controlled)           Opropriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.           Image: the starting work to create awareness to chemical hazards.         Medium         Ist Types of Other Environmental / Equipment Hazards Below           Trenching/excavation         Ladders         Ist Types of Other Environmental / Equipment Hazards Below           Toocks - marine operations         Scaffolding         Environmental / Equipment Hazards           Diving operations         Welding         Environmental / Equipment Hazards:           Forklifts         Roadway work         Medium           Unition activities         Manifts         Environmental / Equipment Hazards:           Forklifts         Roadway work         Medium           Unities         Rairoad work         Energized / Pressurized equip (LOTO)           Overhead/ Underground utilities         Energized / Pressurized equip (LOTO)         Environmental regroup equip (LOTO)           Other         None:		Explosive		Highly toxic		Toluene, Xylene	
Water reactive         Carcinogen         Low         Benzene, B(a)P           DustFumes/ Particulates         Mutagen         Mutagen         None: Mark with an "X"         Particulates           Control the Hazard: (Briefly describe how the identified hazards will be controlled)         None: Mark with an "X"         Particulates           opropriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during aily safety briefings before starting work to create awareness to chemical hazards.         List Types of Other Environmental / Equipmer Hazards Below           Trenching/excavation         Ladders         Poerations         Poerations           Docks - marine operations         Scalfolding         Case Scaling         Poeration gases           Diving operations         Medium         Gas cylinders         Low         Calibration gases           Diving operations work         Railroad work         Medium         Medium         Medium           Water operations work         Railroad work         Energized / Pressurized equip (LO/TO)         Power tools         Drums and containers         Low           Overhead/ Underground utilities         Low         Confined spaces         Drums and containers         Low         Power tools         Order           Onterol the Hazard: (Briefly describe how the identified hazards will be controlled) <td>Chemical Hazards:</td> <td></td> <td></td> <td></td> <td>Low</td> <td>Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene,</td>	Chemical Hazards:				Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene,	
Unstable reactive       Mutagen       Description         DustFumes/ Particulates       Low       None: Mark with an "X"       None: Mark with an "X"         Ontrol the Hazard: (Briefly describe how the identified hazards will be controlled)         porporiate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Medium       Cranes/Hoists/Rigging       List Types of Other Environmental / Equipmer Hazards Below         Trenching/excavation       Ladders       Medium       Cranes/Hoists/Rigging       List Types of Other Environmental / Equipmer Hazards Below         Trenching/excavation       Ladders       Medium       Scaffolding       Docks - marine operations         Docks - marine operations       Welding       Low       Calibration gases         Trinching/excavation       Ladders       Low       Calibration gases         Operations       Welding       Low       Calibration gases         Trins of construction activities       Manifits       Low       Low         Diving operations work       Railroad work       Medium       Medium         Water operations work       Railroad work       Medium       Medium <t< td=""><td>Chemical Hazards:</td><td>Organic peroxide</td><td></td><td>Irritant</td><td>Low</td><td>Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene,</td></t<>	Chemical Hazards:	Organic peroxide		Irritant	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene,	
Particulates       Low         Control the Hazard: (Briefly describe how the identified hazards will be controlled)         opropriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.         Image: the start to construction activities	Chemical Hazards:	Organic peroxide Oxidizer		Irritant Sensitizer		Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene	
Particulates       Control the Hazard: (Briefly describe how the identified hazards will be controlled)         opropriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Image: the starting work to create awareness to chemical hazards.         Image: the starting work to create awareness to chemical hazards.       Imadeing the starting work.       Image: the starting the st	hemical Hazards:	Organic peroxide Oxidizer Water reactive		Irritant Sensitizer Carcinogen		Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene	
porpriate PPE as outlined in the JLA will be used by all on-site employees for each task. In addition, chemical hazards will be discussed daily during ally safety briefings before starting work to create awareness to chemical hazards.           Heavy machinery         Medium         Cranes/Hoists/Rigging         List Types of Other Environmental / Equipmer Hazards Below           Trenching/excavation         Ladders         Medium         Docks - marine operations         Scaffolding         Construction activities         Manlifts         Docks - marine operations         Construction activities         Manlifts         Diving operations         Calibration gases         Construction gases         Calibration gases         Calibration gases         Calibration gases         Calibration gases         Calibration gases         Confined spaces         Diving vork         Calibration gases         Confined spaces         Diver work         Calibration gases         Calibration gases         Calibration gases         Confined spaces         Diver work         Calibration gases         Confined spaces         Diver work         Calibration gases         Calibration gases         Confined spaces         Diver work         Calibration gases         Confined s	hemical Hazards:	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/		Irritant Sensitizer Carcinogen Mutagen		Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene	
Medium     Medium     Medium     Environmental / Equipmer Hazards Below       Trenching/excavation     Ladders        Docks - marine operations     Scaffolding        Ocstruction activities     Manlifts        Diving operations     Welding        Diving operations     Welding        Diving operations work     Readway work     Medium       Vertex     Roadway work     Medium       Water operations work     Railroad work        Heights (fall protection)     Mining work        Overhead/ Underground utilities     High     Energized / Pressurized equip (LO/TO)       Confined spaces     Drums and containers     Low       Power tools     Low     Other       Other     None: Mark with an "X"		Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates		Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X"		Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene	
Medium     Medium     Medium     Environmental / Equipmer Hazards Below       Trenching/excavation     Ladders        Docks - marine operations     Scaffolding        Ocstruction activities     Manlifts        Diving operations     Welding        Diving operations     Welding        Diving operations work     Readway work     Medium       Vertex     Roadway work     Medium       Water operations work     Railroad work        Heights (fall protection)     Mining work        Overhead/ Underground utilities     High     Energized / Pressurized equip (LO/TO)       Confined spaces     Drums and containers     Low       Power tools     Low     Other       Other     None: Mark with an "X"	Control the Hazard: (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider the JLA will be used by all o	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) as for each task. In addition, c	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P	
Docks - marine operations       Scaffolding         construction activities       Manlifts         Diving operations       Welding         Diving operations       Welding         Drilling       Medium         Gas cylinders       Low         Calibration gases         Forklifts       Roadway work         Water operations work       Railroad work         Heights (fall protection)       Mining work         Overhead/ Underground utilities       Energized / Pressurized equip (LO/TO)         Confined spaces       Drums and containers         Power tools       Low         Other       None: Mark with an "X"	Control the Hazard: (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider the JLA will be used by all o arting work to create awarene	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) as for each task. In addition, c hazards.	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P	
operations     Manifts       Diving operations     Welding       Diving operations     Welding       Diling     Medium       Gas cylinders     Low       Calibration gases       Forklifts     Roadway work       Water operations work     Railroad work       Heights (fall protection)     Mining work       Overhead/ Underground utilities     Energized / Pressurized equip (LO/TO)       Confined spaces     Drums and containers       Power tools     Low       Other     None: Mark with an "X"	Control the Hazard: (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider the JLA will be used by all o arting work to create awarene Heavy machinery	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) as for each task. In addition, c hazards.	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen	
Nvironmental/       Construction activities       Manifits       Image: Construction activities         quipment Hazards:       Drilling       Medium       Gas cylinders       Low       Calibration gases         Drilling       Medium       Gas cylinders       Low       Calibration gases         Porklifts       Roadway work       Medium         Water operations work       Railroad work       Medium         Water operations work       Railroad work       Medium         Overhead/ Underground utilities       Energized / Pressurized equip (LO/TO)       Confined spaces         Dower tools       Low       Other       Other         Other       None: Mark with an "X"       Image: Mark with an "X"	Control the Hazard: (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider the JLA will be used by all o arting work to create awarene Heavy machinery Trenching/excavation	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) es for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen	
Diving operations     Welding       Drilling     Medium     Gas cylinders     Low     Calibration gases       prilling     Medium     Gas cylinders     Low     Calibration gases       Water operations work     Railroad work     Medium       Water operations work     Railroad work     Medium       Overhead/Underground utilities     Mining work     Pressurized equip (LO/TO)       Confined spaces     Drums and containers     Low       Power tools     Low     Other       Other     None: Mark with an "X"     Image: Mark with an "X"	<b>Control the Hazard</b> : (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider the JLA will be used by all o arting work to create awarene Heavy machinery Trenching/excavation Docks – marine	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) es for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen	
Drilling       Medium       Gas cylinders       Low       Calibration gases         quipment Hazards:       Forklifts       Roadway work       Medium         Water operations work       Railroad work       Medium         Water operations work       Railroad work       Medium         Overhead/Underground utilities       Mining work       Pressurized equip (LO/TO)         Confined spaces       Drums and containers       Low         Power tools       Low       Other         Other       None: Mark with an "X"       Difference	Control the Hazard: (B	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/ Particulates           rriefly describe how the ider           arting work to create awarene           Heavy machinery           Trenching/excavation           Docks – marine operations	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) s for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen	
Forklifts       Roadway work       Medium         Water operations work       Railroad work       Medium         Water operations work       Railroad work       Image: Construct of the space of	<b>Control the Hazard</b> : (B	Organic peroxide Oxidizer Water reactive Unstable reactive Dust/Fumes/ Particulates riefly describe how the ider In the JLA will be used by all o arting work to create awarene Heavy machinery Trenching/excavation Docks – marine operations Construction activities	ntified hazards	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) es for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen	
Water operations work       Railroad work         Heights (fall protection)       Mining work         Overhead/ Underground utilities       Energized / Pressurized equip (LO/TO)         Confined spaces       Drums and containers         Power tools       Low         Other       Other         Other       None: Mark with an "X"	ontrol the Hazard: (B opropriate PPE as outlined in ally safety briefings before st	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/           Particulates           riefly describe how the ider           the JLA will be used by all o           arting work to create awarene           Heavy machinery           Trenching/excavation           Docks – marine           operations           Diving operations	ntified hazards n-site employee sss to chemical Medium	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) s for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding	Low hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
Overhead/ Underground High       Energized / Pressurized equip (LO/TO)         Confined spaces       Drums and containers         Power tools       Low         Other       None: Mark with an "X"         Control the Hazard: (Briefly describe how the identified hazards will be controlled)	pontrol the Hazard: (B poropriate PPE as outlined ir ily safety briefings before sta	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/ Particulates           riefly describe how the ider           the JLA will be used by all o           arting work to create awarene           Heavy machinery           Trenching/excavation           Docks – marine           operations           Construction activities           Diving operations           Drilling	ntified hazards n-site employee sss to chemical Medium	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) s for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
utilities     High equip (LO/TO)       Confined spaces     Drums and containers       Power tools     Low       Other     Other       Other     None: Mark with an "X"	ontrol the Hazard: (B opropriate PPE as outlined ir ally safety briefings before sta	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/           Particulates   riefly describe how the ider In the JLA will be used by all o arting work to create awarene Heavy machinery Trenching/excavation Docks – marine operations Construction activities Diving operations Diving operations Diving operations Diving operations Diving operations Diving water operations work	ntified hazards n-site employee sss to chemical Medium	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) es for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders Roadway work Railroad work	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
Confined spaces         Drums and containers         Low           Power tools         Low         Other         Other           Other         None: Mark with an "X"         Other	Control the Hazard: (B	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/ Particulates           riefly describe how the ider           the JLA will be used by all o           arting work to create awarene           Trenching/excavation           Docks - marine           operations           Construction activities           Diving operations           Drilling           Forklifts           Water operations work           Heights (fall protection)	ntified hazards n-site employee sss to chemical Medium	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) s for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders Roadway work Railroad work Mining work	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
Power tools         Other         Other           Other         None: Mark with an "X"         Image: Control the Hazard: (Briefly describe how the identified hazards will be controlled)	Control the Hazard: (B ppropriate PPE as outlined ir aily safety briefings before st	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/           Particulates           riefly describe how the ider           the JLA will be used by all o           arting work to create awarene           Water reactive           Unstruction           Ocks – marine           operations           Construction activities           Diving operations           Drilling           Forklifts           Water operations work           Heights (fall protection)           Overhead/ Underground	ntified hazards n-site employee sss to chemical Medium Medium	Irritant Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) es for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders Roadway work Railroad work Mining work Energized / Pressurized	Low	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
Other None: Mark with an "X" Other O	Control the Hazard: (B ppropriate PPE as outlined ir aily safety briefings before st	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/ Particulates           riefly describe how the ider           the JLA will be used by all o arting work to create awarene           Heavy machinery           Trenching/excavation           Docks – marine operations           Diving operations           Drilling           Forklifts           Water operations work           Heights (fall protection)           Overhead/ Underground utilities	ntified hazards n-site employee sss to chemical Medium Medium	Irritanit Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) so for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders Roadway work Railroad work Mining work Energized / Pressurized equip (LO/TO)	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
	Control the Hazard: (B ppropriate PPE as outlined ir aily safety briefings before st	Organic peroxide           Oxidizer           Water reactive           Unstable reactive           Dust/Fumes/           Particulates           riefly describe how the ider           the JLA will be used by all o           arting work to create awarene           Water reactive           Trenching/excavation           Docks - marine           operations           Drilling           Forklifts           Water operations work           Heights (fall protection)           Overhead/ Underground           utilities           Confined spaces	Attified hazards n-site employee ss to chemical Medium Medium High	Irritanit Sensitizer Carcinogen Mutagen None: Mark with an "X" will be controlled) ss for each task. In addition, c hazards. Cranes/Hoists/Rigging Ladders Scaffolding Manlifts Welding Gas cylinders Roadway work Railroad work Mining work Energized / Pressurized equip (L0/TO) Drums and containers	hemical haza	Benzene, B(a)P, Fluorene, Ethylbenzene, Naphthalene, Toluene, Xylene Benzene, B(a)P ards will be discussed daily during List Types of Other Environmental / Equipmen Hazards Below	
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	Animal/Human fluids or blood		Contaminated Needles		List Types of Other Biologica Hazards Below
Biological Hazards	Animal/Human tissue(s) Poisonous/irritating		Live Bacterial Cultures		
	plants		Insects/rodents/snakes		
	Other:		None: Mark with an "X"	Х	
Control the Hazard: (Brie	fly describe how the ider	ntified hazards	will be controlled)		
	,				
	D ee e				
	Repetitive motion	Low	Limited movement		List Types of Other Ergonomic Hazards Below
<b>F</b> amera and a 11a and a	Awkward position		Forceful exertions		
Ergonomic Hazards	Heavy lifting	Medium	Vibration		
	Frequent lifting Other:	Low	Other:		
			None: Mark with an "X"		
Control the Hazard: (Brie					
Use buddy system during hand o	ligging activities to minimiz	e back strain. 1	ake breaks as needed. Use tv	vo people to	lift objects greater than 50
oounds.					
	Personal safety		Employees working		List Types of Other Persona
	,	Low	early/late	Low	Safety / Security Hazards
	<b>0</b>				Below
	Security issue		Potentially dangerous wildlife		
Personal Safety/Security	Project site in isolated		Guard or stray dogs in area		
	area				
	Employees working alone	Low	No/limited cell phone service		
	Fatigue	Low	Other:		
	Other		None: Mark with an "X"		
Control the Hazard: (Brie	fly describe how the ider	ntified hazards	will be controlled)		
Employees will take breaks as no				uring and at	the end of the day
Employees will take breaks as in	couce to avoid langue and			and at	the end of the day.
		Low		Medium	List Types of Other Driving
	Driving early/late		City driving		Hazards Below
	Driving long trips		Pulling a trailer		
Driving Safety	Driving long trips Driving off-road		Pulling a trailer ATV driving:		
Driving Safety	Driving off-road Bad weather driving	Medium	ATV driving: Other		
Driving Safety	Driving off-road Bad weather driving Other		ATV driving: Other None: Mark with an "X"		
	Driving off-road Bad weather driving Other		ATV driving: Other None: Mark with an "X"		
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# **ARCADIS**

Appendix C

Job Loss Analyses

### General

Client Name	ARCADIS-AGMI
JSA ID	15
Job Name	General Industry-Driving - passenger vehicles
Task Description	Driving Passenger Vehicle or Pick Up Truck
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	1/29/2009 2:10:18 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	1/31/2009	1/29/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/1/2009	1/29/2009		Leichner III, Charles	True
HASP Reviewer	Coppola, Mija	1/31/2009	1/29/2009	True	Leichner III, Charles	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	01/29/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Preforming Pre-trip inspections	1	Cuts scrapes to hands and fingers checking engine fluids	Use TRACK to plan inspection activity in the engine compartment. Wear protective gloves if reaching in poorly illuminated areas of the engine.	
		2	Pinch crush hazards to hands and fingers checking engine fluids or closing doors.	Identify and keep hands fingers away from pinch hazards from doors and vehicle hood or tailgate (if present).	
		3	Awkward body positions checking tires, spare tire, undercarriage, or engine compartment.	Maintain neutral body positions and avoid awkward reaches under the vehicle or in engine compartment.	
		4	Failure to inspect vehicle emergency equipment may result in extensive vehicle damage or delay treatment in the event of injury	Conduct equipment inspections by visibly inspecting fire extinguisher and first aid kit for cleanliness, in date items/tags, readiness for use.	
2	Vehicle loading and unloading	1	Object placement obstructing rear, side or blindspot view	Avoid placing objects in a manner that obstructs your view, brake equipment down to a smaller more manageable size to keep low profile in vehicle. If hanging clothes in vehicle, place in manner that does not obstruct blind spots.	

2	Vehicle loading and unloading	L	Unsecure objects causing pedal, steering or gear shift obstruction or injury during vehicle operation.	Secure all loads in vehicle (both in the bed of trucks and in passenger cabin) to prevent unanticipated movement or shifting that could injure driver, passenger, or affect safe operation of vehicle.	
		3	Obstuction of vehicle safety equipment caused by object placment in vehicle.	Keep emergency equipment clear and unobstructed to ensure ready availablity.	
3	Vehicle operation	1	Failure to use Smith System "5- Keys" increases risk of accident and injruy.	Use Smith System "5-Keys", maintain space cushion around vehicle, maintain 4 second rule and add (second for each addtional hazard (wet roads, snow, etc). Brake gradual, keep eyes moving, check mirrors every 6-8 seconds, use turn signals, focus on relavent objects, use early lane positioning when approaching turns.	
		2	Injury or death from failure to wear seatbelt	Always wear seatbelts even if driving short distances off of a public roadway.	
		3	Cell phone use increases risk of accidnt and injury	Avoid using cell phones in any capacity when operating a vehicle, check client for cell use on project sites and follow requirements. Follow all local laws.	
		4	Use of radar detectors encourages speeding resulting in increased risk for accident or injury	Use of radar detectors and similar devices is prohibited.	
4	Routine maintenance	1	Pinch crush hazards to hands and fingers replacing engine fluids or closing doors/hood.	Inspect and indetify pinch and crush hazards and keep hands/fingers clear when closing hood, tailgates, or doors.	
		2	Burn hazards to hand form checking/replacing fluids in engine compartment	When practical allow engine to cool prior to servicing or adding fluids. Use protective gloves.	
		3	Vehicle damage from improper fuse replacement	Never replace a fuse with a higher amperage than the one being replaced. Only replace fuses of type being replaced.	
		4	Failing to use Wright Express for vehicles equipped with fuel card impairs maintenace tracking that could affect vehicle safety	If vehicle is assigned a Wright Express Card, use the card so accurate maintenance tracking can be performed by LeasePlan.	

Туре	Supply	Description	Required
Communication Devices	mobile phone		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Traffic Control	Other	Roadway emergency kit	Required

### General

N.	
Client Name	ARCADIS-AGMI
JSA ID	43
Job Name	General Industry-Site inspection/walkover - building
Task Description	walkover-building
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	2/3/2009 7:03:50 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	2/5/2009	2/3/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/6/2009	2/3/2009		Leichner III, Charles	True
HASP Reviewer	Coppola, Mija	2/5/2009	2/2/2009	True	Leichner III, Charles	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	02/02/2009	

### Job Steps

ob tep	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Evaluate site upon arrival for personal safety and security	1	Building could have structural issues creating fall hazards, or debris could cause slip/trip/fall hazards.	Assess building, and make sure that hazards were similar as what was scoped in the project or reported by the client. Additional hazards that could impact safety of personnel should be called into the project manager. An engineer should perform an assessment on any building that appears structurally unsafe.	
		2	personal security	Assess potential personal security issues prior to starting work. Verify cell phone reception. In high risk areas, have a security escort as needed. Notify PM/TM or supervisor of time of entry and anticipated time of exit. Inform that person of anticipated walking route.	
2	Building inspection	1	Limited lighting and visibility could cause slip/trip/falls over hard to see hazards on the ground	All personnel will carry their own flashlight. Carry extra batteries.	
		2	unstable or slippery (oil and ice covered) walking and work surfaces	Use caution and proper footwear with traction for potential slippery surfaces. Walk around these areas when possible.	
		3	Inadequate barricades and guards around pits or depressions could cause fall or trip hazard.	Use the flashlight to clear the area prior to entry, walk slowly and do not walkthrough puddles as it may be a larger depression filled with water/oil.	
		4	PCB containing oil, residual chemicals could be encountered	Avoid all dermal contact with equipment/chemicals/etc located within the building. Wear nitrile gloves when touching or picking up objects left in the building.	
		5	Potential asbestos containing material could be encountered, especially in buildings constructed prior to 1987.	Avoid disturbing material that could potentially contain asbestos. This includes pipe and boiler insulation, transite board, ceiling tiles, and floor tiles. If damaged material is observed, avoid the area and disturbing the material.	
		6	Limited access and egress to the building	Confirm that everyone entering the building is aware of the exit route and periodically discuss where the point of exit is in relation to current location as you move throughout the building.	
		7	Stray animals, mice, rats	Make lots of noise while traveling through the building and carry repellent spray in the event of encountering stray animals. If a dangerous or aggravated animal is spotted, leave the building immediately and contact animal control.	
3	Roof inspections, or elevated heights inspections	1	Personal injury could occur from elevated falls	Follow the JLA for Elevated Heights	

### Personal Protective Equipment

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	boots		Required
Head Protection	hard hat		Required

Туре	Supply	Description	Required
Communication Devices	mobile phone		Required
Miscellaneous	fall protection (specify type)		Required
Miscellaneous	Other	flashlight	Required

#### General

Client Name	ARCADIS-AGMI
JSA ID	21
Job Name	General Industry-Roadway work
Task Description	Roadway work
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	1/29/2009 8:13:32 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	1/31/2009	1/29/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/1/2009	1/29/2009		Leichner III, Charles	True
HASP Reviewer	Coppola, Mija	1/31/2009	1/29/2009	True	Leichner III, Charles	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	01/29/2009	
Quality Reviewer	Coppola, Mija	Quality	01/30/2009	test

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Deployment and removal of traffic control devices	1	Lifting hazards and awkward body positions from moving warning signs and control devices	Avoid excessive force pushing or pulling devices from vehicle; use the buddy system for heavier items; lift with legs and not back; avoid lifting and twisting motions.	ARCADIS H&S Handbook section III LL
		2	Struck by vehicle during placement	Wear high visibility clothing and Class II (minimum) traffic vest. Choose lime green color to avoid motorist confusion with traffic barrels. Always face oncoming traffic, use spotter if performing work that keeps focus off traffic. Ensure vehicle equipped with light bars and/or other warning devices and ensure they are activated, including vehicle flashers.	ARCADIS H&S Handbook section III LL
		3	Increased risk of injury (ergonomic from reacted moving or impact from increased vehicle exposure) from poor traffic control planning and implementation	Develop traffic control plan consistent with Manual of Uniform Traffic Control Devices, ensure lane closure tapers are computed properly, place devices in a manner that offers protection as other devices are deployed, place early warning devices first to warn drivers of pending work zone.	ARCADIS H&S Handbook section III LL

2	Flagger activities	1	Struck by vehicle while performing activity	Always face oncoming traffic, wear high visibility clothing described in step 1 above. Flaggers to be properly trained in proper flagging technique, if using paddles, ensure correct paddle warning displayed.	Certain states require flagger training: www.flagger.com
		2	Fatigue form standing in one position for extended periods of time.	Use job rotation when practical, shift weight form one leg to the other periodically, wear comfortable boots.	Certain states require flagger training: www.flagger.com
		3	Dehydration, heat stress (summer months), cold stress (winter months), sunburn, windburn	Ensure drinking water is in immediate vicinity of the flagger, check with flagger periodically to evaluate signs of heat or cold stress, avoid caffeine or sugary drinks during hot or cold weather, schedule work for worker to eat at regular intervals, wear sun block	Certain states require flagger training: www.flagger.com
		4	Struck by debris off roadway from passing vehicles	Be aware of hazard and be vigilant for debris, wear eye protection at all times.	Certain states require flagger training: www.flagger.com
3	Working in work zone	1	Struck by vehicle while performing work	Always stay behind protective barriers or channeling devices, never park vehicle that exposes workers to on coming traffic outside of barriers and channeling devices. Wear clothing and PPE described in step one above. Park vehicles within work zone to act as barriers to oncoming traffic when possible.	
		2	Struck by equipment in work area	Establish eye contact with all equipment operators when entering equipment operating radius, wear high visibility clothing and PPE as described in step one above. Park project vehicle away form active work are but still in work zone barriers or channeling devices.	
		3	Slips, trips and falls on wet or uneven surfaces in road right of way.	Wear proper footwear with good tread and ankle support. Plan route when walking on sloped surfaces, when walking along roadway stay as far off roadway as possible to avoid falling into traffic if tripping.	

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required
Miscellaneous PPE	traffic vestClass II or III		Required

Туре	Supply	Description	Required
Communication Devices	mobile phone		Required
Communication Devices	walkie talkie	if using flaggers	Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Traffic Control	traffic cones		Required

### General

Client Name	ARCADIS-AGMI
JSA ID	346
Job Name	Environmental-Air knife/hydro knife
Task Description	air knife/hydro knife
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	5/29/2009 12:12:21 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	5/29/2009	5/29/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	5/29/2009	5/29/2009		Leichner III, Charles	True
HASP Reviewer	Moyers, Samuel	6/12/2009	6/15/2009	True	Coppola, Mija	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Moyers, Samuel	Approve	06/15/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Check and clear proposed hydro-knife locations for the presence of underground and overhead utilities	1	Staff can be hit by vehicular traffic	Wear reflective traffic vest. Establish work zone with cones.	Utility Clearance H&S Procedure: ARCHSFS019
		2	Underground utilities can be encountered	Follow ARCADIS policy on utility location	Utility Clearance H&S Procedure: ARCHSFS019
2	Clear hole using the hydro-knife	1	Subsurface could have material that may contain rocks/sharp objects. Flying debris could cause injury to eyes, face, arms and legs; Water spray could contain mud, sharp debris or chemicals of concern;	Stay back a minimum of five from the hydro-knife while in operation by the contractor. Wear safety glasses, leather gloves, hardhat.	
		2	operation of the hydro-knife generates excessive noise	Hearing protection is required when the equipment is in operation	
		3	vacuum unit has a large amount of suction	do not put any part of your body near the end of the hose	
3	Barricade open holes	1	Holes can be difficult to see depending on their size, and site workers could twist their ankle or fall if they step on an open hole.	Holes can be as large as 6-8 inches in diameter and as deep as 7 feet. Heavy cones, orange barrels or cones with caution tape should be used to protect the holes	
		2	Lifting hazards from carrying heavy cones or orange barrels	Minimize number of cones lifted at one time. Use team lift approach when practicable.	

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required
Hearing Protection	ear plugs		Required
Miscellaneous PPE	traffic vestClass II or III		Required

Туре	Supply	Description	Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)		Required
Traffic Control	traffic cones		Required

#### General

N.	
Client Name	ARCADIS-AGMI
JSA ID	44
Job Name	Environmental-Drilling, soil sampling, well installation
Task Description	drilling with drill rig
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	2/4/2009 7:25:55 AM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	2/6/2009	2/4/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/7/2009	2/4/2009		Leichner III, Charles	True
Developer	Moyers, Samuel	2/7/2009	2/4/2009		Coppola, Mija	True
HASP Reviewer	Coppola, Mija	2/6/2009	2/2/2009	True	Leichner III, Charles	True

**Reviewer Comments** 

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	02/02/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Set up necessary traffic and public access controls	1	Struck by vehicle due to improper traffic controls	Use a buddy system for placing site control cones and/or signage. Position vehicle so that you are protected from moving traffic. Wear Class II traffic vest	
2	Utility Clearance	1	Potential to encounter underground or aboveground utilities while drilling	Complete utility clearance in accordance with the ARCADIS H&S procedure	ARCADIS H&S Procedure ARCHSFS019
3	General drill rig operation	1	Excessive noise is generated by rig operation.	When the engine is used at high RPMs or soil samples are being collected, use hearing protection.	
		2	During drill rig operation, surfaces will become hot and cause burns if touched, and COCs in the soils more readily vaporize generating airborne contaminates.	Due to friction and lack of a drilling fluid, heat will be produced during this method. Mainly drill augers. Be careful handling split spoons. Wear proper work gloves. When soils and parts become heated, the COC could volatilize. Air monitoring should always be performed in accordance with the HASP.	

3	General drill rig operation	3	Moving parts of the drilling rig can pull you in causing injury. Pinch points on the rig and auger connections can cause pinching or crushing of body parts.	Stay at least 5 feet away from moving parts of the drill rig. Know where the kill switch is, and have the drillers test it to verify that it is working. Do not wear loose clothing, and tie long hair back. Avoid wearing jewelry while drilling. Cone off the work area to keep general public away from the drilling rig	
		4	Dust and debris can cause eye injury and soil cuttings and/or water could contain COCs.	Wear safety glasses and stay as far away from actual drilling. W operation as practicable. Wear appropriate gloves to protect from COCs.	
		5	Drilling equipment laying on the ground (i.e. augers, split spoons, decon equipment, coolers, etc), create a tripping hazard. Water from decon buckets generate mud and cause a slipping hazard.	Keep equipment and trash picked up, and store away from the primary work area.	
		6	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
4	Mudd rotary drilling	1	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
		2	This technology uses fluid, which collects with sediments in large basin. Fluid can splash out and cause slipping/mud hazard. Liquid mixture can splash into your eyes.	Wear rubber boots if needed, and keep clear of muddy/wet area as much as practicable. If area becomes excessively muddy, consider mud spikes or covering the area with a material that improves traction. Wear safety glasses.	
5	Hollow stem auger drilling	1	All hazards in step 3 apply. Additionally,The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
6	Air rotary drilling	1	this drilling method works with high air pressure and can generate flying debris that can strike your body or get debris in your eyes.	When the drill rig is being driven into media, it will produce flying debris. The flaps behind the drill rig should stay closed whenever possible to reduce the risk of flying debris. Safety glasses and hard hat should always be worn when the drill rig is operating. When penetrating asphalt protect surrounding cars that may be present to avoid debris damage to paint or winshields.	
		2	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
		3	When drilling through bedrock prior to groundwater dust can be produced from pulverization. Inhalation of dusts/powder can occur	Supplemental water should be used to manage dust creation and/or dust masks if necessary.	

7	Reverse rotary drilling	1	This method will use fresh water to pump out drill cuttings through the center of the casing. Water/sediment mixture is generated and could cause contact with impacted soils or groundwater	Ensure the pit construction can hold the amount of cuttings that are anticipated. Air monitoring should also be used of pit area	
		2	Fire hydrants are often used for water source. Hydrants deliver water at high pressure. Pressurized water can cause flying parts/debris and excessive slipping hazards.	Water usage from fire hydrants should be cleared with local muncipalities prior to use. Only persons that know how to use the hydrant should be performing this task. Ensure all connections are tight, and hose line is not run over to cut by traffic. Any leaks from the hydrant should be reported immediately.	
		3	Settling pit construction can cause tripping hazard from excavated soils, and plastic sheeting can cause slipping.	cone off the area to keep the general public/visitors away from the settling pit. Ensure proper sloping of excavation.	
		4	The raised derrick can strike overhead utilities, tree limbs or other elevated items	Never move the rig with the derrick up. Ensure there is proper clearance to raise the derrick, and that you are far enough away from overhead power lines. See the Utility Location H&S policy and procedure for guidance.	
8	Rotosonic drilling	1	Fire hydrants are often used for water source. Hydrants deliver water at high pressure. Pressurized water can cause flying parts/debris and excessive slipping hazards.	Water usage from fire hydrants should be cleared with local muncipalities prior to use. Only persons that know how to use the hydrant should be performing this task. Ensure all connections are tight, and hose line is not run over to cut by traffic. Any leaks from the hydrant should be reported immediately	
		2	This method requires a lot of clearance. The drill head can turn 90 degrees to attach to the next drill flight or casing. This usually requires a large support truck to park directly behind the rig. As the drill head raises the new casing flight is angled down at the same time until it can be turned completely vertical.	Ensure sufficient overhead clearance.	
		3	Heavy lifting of cores can cause muscle strain.	Always use 2 people to move core containers. Use caution moving core samples to layout area. Plan layout area to ensure adequate aisle space between core runs for logging. Keep back straight and use job rotation.	
		4	The rotosonic drill head can move very quickly up and down while working on a borehole. Moving parts can strike someone or catch body parts	The operator and helper must communicate and stay clear of the path of the drill head. The drill utilizes two large hydrualic clamps to continuously hold casings while load/unloading previous casings. Do not wear loose clothing.	
9	Direct push drilling	1	The drill rods will be handled by workers most of the time rather than the rig doing it, therefore pinch points can cause lacerations and crushing of fingers/body parts.	Keep a minimum of 5 feet away from drill rig operation and moving parts.	

9	Direct push drilling	2	The direct push rigs are uaually meant to fit in spaces where larger rig can't. Tight spaces can pin workers.	Do not put yourself between the rig and a fixed object. Use Spotters or a tape measure to ensure clearances in tight areas. Pre-plan equipment movement from one location to the next.	
		3	some direct push equipment is controlled by wireless devices. These controls can fail and equipment can strike workers or cause damage to property.	The drill rig should be used in a large open area to test wireless controls prior to moving to boring locations. The operator of the rig will test the kill switch with wireless remote prior to use. Operator will stay in range of rig while moving so that wireless signal will not be too weak and cause errors to the controls.	
		4	Sampling sleeves must be cut to obtain access to soil. Cutting can cause lacerations.	Preferably let the driller cut the sleeves open. Many drillers have holders for the sleeve to allow for stability when cutting. If we cut the sleeves, use a hook blade, change blade regularly, and cut away from the body.	
10	Rock Coring	1	flying debris can hit workers or cause debris to get in eyes.	Rock chips or overburden may become airborne from drilling method. Wear safety glasses and hard hat and remain at a safe distance from back of drill rig.	
		2	Heavy lifting of cores can cause muscle strain.	Always use 2 people to move core containers. Use caution moving core samples to layout area. Plan layout area to ensure adequate aisle space between core runs for logging. Keep back straight and use job rotation.	
11	Sample collection and processing	1	Injuries can result from pinch points on sampling equipment, and from breakage of sample containers.	Care should be taken when opening sampling equipment. Look at empty containers before picking them up, and do not over-tighten container caps. Use dividers to store containers in the cooler so they do not break.	Sample cooler handling JLA
		2	lifting heavy coolers can cause back injuries	Use two people to move heavy coolers. Use proper lifting techniques.	Sample cooler handling JLA
12	Monitoring well installation	1	Same hazards as in Step 3 with general drill rig operation	See step 3	
		2	monitoring well construction materials can clutter the work area causing tripping hazards.	Well construction materials should be picked up during the well installation process.	
		3	Heavy lifting can cause muscle strains, and cutting open bags can cause lacerations.	Well construction materials are usually 50 lbs or greater. Team lift or use drill rig to hoist bags. Always use work gloves while cutting open bags.	
		4	Well pack material (i.e. sand, grout, bentonite) can become airborne and get in your eyes.	Wear safety glasses for protection from airborne sand and dust.	
		5	Cutting the top of the well to size can cause jagged/sharp edges on the top of the well casing.	Wear gloves when working with the top of the well casing, and file any sharp jagged edges that resulted from cutting to size.	
13	Soil cutting and purge water management	1	Moving full drums can cause back injury, or pinching/crushing injury.	Preferably have the drilling contractor move full drums with their equipment. If this is not practicable, use lift assist devices such as drum dollys, lift gates, etc. Employ proper lifting techniques, and perfrom TRACK to identify pinch/crush points. Wear leather work gloves, and clear all walking and work areas of debris prior to moving a drum.	Drum handling JLA

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	chemical resistant gloves (specify type)		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required
Hearing Protection	ear plugs		Required
Miscellaneous PPE	traffic vestClass II or III		Required
Respiratory Protection	dust mask		Recommended

Туре	Supply	Description	Required
Communication Devices	mobile phone		Required
Decontamination	Decon supplies (specify type)		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)	bottle	Required
Traffic Control	traffic cones		Required

### General

Client Name	ARCADIS-AGMI
JSA ID	45
Job Name	Environmental-Groundwater Sampling and free product recovery
Task Description	groundwater sampling
Project Number	00000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	2/4/2009 9:12:19 AM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	2/6/2009	2/4/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/7/2009	2/4/2009		Leichner III, Charles	True
HASP Reviewer	Coppola, Mija	2/6/2009	2/6/2009	True	Leichner III, Charles	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	02/06/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Ùcæť∧ÁæcÁ,¦∧Ëâ∧c∿¦{āj∧å •æ{] āj*Á{(&cæa∄}} æ}åÁ*∧cÁ]Á{[¦\Á[}^Áæ)å •æ{] āj*Á <sup>××</sup> āj{^}c	1	personnel could be hit by vehicluar traffic.	Set-up cones and establish work area. Position vehicle so that field crew is protected from site traffic. Unload as close to work area as safely possible.	
		2	Sampling equipment, tools and monitoring well covers can cause tripping hazard	Keep equipment picked up and use TRACK to assess and changes	
2	Open wells to equilibrate and gauge wells	1	When squatting down, personnel can be difficult to see by vehicular traffic.	Wear Class II traffic vest if wells are located proximal to vehicular traffic. Use tall cones and the buddy system if practicable.	
		2	pinchpoints on well vault can pinch or lacerate fingers	Use correct tools to open well vault/cap. Wear leather gloves when removing well vault lids, and chemical protective gloves while guaging. Wear proper PPE including safety boots, knee pads and safety glasses.	
		3	Lifting sampling equipment can cause muscle strain	Unload as close to work area as safely possible; use proper lifting and reaching techniques and body positioning; don't carry more than you can handle, and get help moving heavy or awkward objects.	

2	Open wells to equilibrate and gauge wells	4	Pressure can build up inside well causing cap to release under pressure	Keep head away from well cap when removing. If pressure relief valves are on well use prior to opening well	
3	Ó^*ājÁÚ≚¦*āj*ÁY^∥Áænjå Ô[∥^&cāj* Úæiæ{{^c^¦ÁT^æne`¦^{ ^}om	1	Electrical shock can occur when connecting/disconnecting pump from the battery.	Make sure equipment is turned off when connecting/disconnecting. Wear leather gloves. Use GFCIs when using powered tools and pumps. Do not use in the rain or run electrical cords through wet areas.	
		2	purge water can spill or leak from equipment	Stop purging activities immediately, stop leakage and block any drainage grate with sorbent pads. Call PM to notify them of any reportable spill.	
		3	Water spilling on the ground can cause muddy/slippery conditions	Be careful walking in work area when using plastic around well to protect from spillage	
		4	lacerations can occur when cutting materials such as plastic tubing	When cutting tubing, use tubing cutter. No open fixed blades should ever be used. When possible wear work gloves, leather type.	
		5	purge water can splash into eyes	Pour water slowly into buckets/drums to minimize splashing. Wear safety glasses	
4	Collect GW or Free Product Sample	1	Working with bailer rope can cause rope burns on hands.	Slowly raise and lower the rope or string for the bailer. Wear appropriate gloves for the task.	
		2	sample containers could break or leak preservative	Discard any broken sampleware or glass properly. Do not overtighten sample containers. Wear chemical protective gloves	
5	Recovery of Free Product from well	1	exposure to free product	Additional chemical protection may be necessary based on the type of product. Additionally, safety goggles, a faceshield, or respiratory protection may be required. Verify in the HASP.	
6	Staging of Well Purge water and/or Free Product	1	Muscle strains can occur when moving purge water or drums	If using buckets, do not fill buckets up to the top. Always keep lid on buckets when traveling or moving them to another location. Only half fill buckets so when dumping the buckets weigh less. See drum handling JLA for movement of drums.	Drum handling JLA

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	chemical resistant gloves (specify type)		Required
Hand Protection	work gloves (specify type)	leather	Required
Head Protection	hard hat		Required

Туре	Supply	Description	Required
Communication Devices	mobile phone		Required
Decontamination	Decon supplies (specify type)		Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)	bottle	Required
Traffic Control	traffic cones		Required

### General

Client Name	ARCADIS-AGMI
JSA ID	166
Job Name	Environmental-Sample cooler handling
Task Description	Sample cooler handling
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	5/1/2009 8:07:23 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	5/11/2009	5/11/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	5/11/2009	5/11/2009		Leichner III, Charles	True
HASP Reviewer	Moyers, Samuel	5/25/2009	5/13/2009	True	Coppola, Mija	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Moyers, Samuel	Revise		Kevlar is required? Leather work gloves are listed. i suggest just leather gloves.
HASP Reviewer	Moyers, Samuel	Approve	05/13/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Transfer field samples to sample packing area	1	Lifting heavy coolers may result in muscle strain especially to lower back.	Use proper lifting techniques and keep back straight. Use buddy system for large coolers, Use mechanical aids like hand trucks if readily available to move coolers. Do not over fill coolers with full sample containers for temporary movement to the sample prep area. Ensure an adequate supply of sample coolers are in field	
		2	Hazards to hands from broken glass caused by over tightening lids or improper placement in cooler	Inspect all bottles and bottle caps for cracks/leaks before and after filling container. Do not over tighten sample lids. Clean up any broken bottles immediately, avoid contact with sample preservatives. Wear leather gloves when handling broken glass.	
		3	Exposure to chemicals ( acid preservatives or site contaminants) on the exterior of sample bottles after filling.	Wear protective gloves for acid preservatives and safety glasses with side shields during all sample container handling activities (before and after filling), Once filled follow project specific HASP PPE requirements for skin and eye protection.	

1	Transfer field samples to sample packing area	4	Samples containing hazardous materials may violate DOT/IATA HazMat shipping regulations	All persons filling a sample bottle or preparing a cooler for shipment must have complete ARCADIS DOT HazMat shipping training. Compare the samples collected to the materials described in the Shipping Determination for the Project and ensure consistent. Re perform all Shipping determinations if free product is collected and not anticipated during planning.	
2	Sample cooler selection	1	Sample coolers with defective handles, lid hinges, lid hasps cracked or otherwise damaged may result in injury (cuts to hands, crushing of feet if handle breaks etc)	Only use coolers that are new or in like new condition, No rope handled coolers unless part of the manufacturer's handle design.	ARCADIS Shipping Guide US-0
		2	Selection of excessively large coolers introduces lifting hazards once the cooler is filled.	Select coolers and instruct lab to only provide coolers of a size appropriate for the material being shipped. For ordinary sample shipping sample coolers should be 48 quart capacity or smaller to reduce lifting hazards.	ARCADIS Shipping Guide US-0
3	Pack Samples	1	Pinch points and abrasions to hands from cooler lid closing unexpectedly	Beware that lid could slam shut; block/brace if needed; be wary of packing in strong winds. New coolers may be more prone to self closing, tilt cooler back slightly to facilitate keeping lid open.	
		2	Awkward body positions and contact stress to legs and knees when preparing coolers on irregular or hard ground surfaces.	Plan cooler prep activities. Situate cooler where neutral body positions can be maintained if practical, like truck tailgate. Avoid cooler prep on gravel rough surfaces unless knees and legs protected during kneeling.	
		3	Frostbite or potential for oxygen deficiency when packing with dry ice. Contact cold stress to fingers handling blue ice or wet ice	Dry ice temperature is -109.30F. Wear thermal protective gloves. DO NOT TOUCH with bare skin! Dry ice sublimates at room temp and could create oxygen deficiency in closed environment. Maintain adequate ventilation! Do not keep dry ice in cab of truck. Wear gloves when handling blue ice or gaging wet ice. Dry Ice is DOT regulated for air shipping, follow procedures in Shipping Determination.	
4	Sealing, labeling and Marking Cooler	1	Cuts to hands and forearms from strapping tape placement or removing old tape and labels	Do not use a fixed, open-blade knife to remove old tags/labels, USE SCISSORS or other safety style cutting device. Only use devices designed for cutting. Do not hurry through task.	
		2	Lifting and awkward body position hazards from taping heavy coolers, dropping coolers on feet during taping.	Do not hurry through the taping tasks, ensure samples in cooler are evenly distributed in cooler to reduce potential for overhanging cooler falling off edge of tailgate/table when taping.	
		3	Improper labeling and marking may result in violation of DOT/IATA Hama shipping regulations delaying shipment or resulting in regulatory penalty	Do not deviate from ARCADIS Shipping Guide or Shipping Determination marking or labeling requirements.	
5	Offering sample cooler to a carrer or lab couriour for shipment.	1	Lifting heavy coolers may result in muscle strain especially to lower back.	See lifting hazard controls above.	

5 Offering sample cooler to a carrer or lab couriour for shipment.	2	HazMat shipping regulations.	Promptly report all rejected and refused shipments to the ARCADIS DOT Program Manager. Do Not re- offer shipment if carrier requires additional labels markings or paperwork inconsistent with your training or Shipping Determination without contacting the ARCADIS DOT Compliance Manager.	
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Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Hand Protection	chemical resistant gloves (specify type)	nitrile	Required
Hand Protection	work gloves (specify type)	leather	Required

Туре	Supply	Description	Required
Miscellaneous	Other	Scissors	Required

#### General

Client Name	ARCADIS-AGMI
JSA ID	38
Job Name	General Industry-Surveying - land
Task Description	land surveying
Project Number	00000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status Name	(3) Completed
Cretaion Date	2/2/2009 12:26:58 PM

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	2/4/2009	2/2/2009		Leichner III, Charles	True
Developer (Primary Contact)	Coppola, Mija	2/5/2009	2/2/2009		Leichner III, Charles	True
HASP Reviewer	Coppola, Mija	2/4/2009	2/2/2009	True	Leichner III, Charles	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	02/02/2009	

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Site reconnaissance and walk- around	1	Slips/trips/falls can occur from walking on uneven ground surface.	Survey the site upon arrival. Note any site conditions that may pose a potential hazard.	RŠŒËŪ[æå,æੰÁY[¦∖ ŒÜÔPÙØŬ€FÏ
		2	Site workers or equipment can be struck by site vehicular traffic	Wear Class II traffic vest and cone off the work area. Follow the JLA and Field H&S Handbook for roadway work.	RŠŒËÜ[æå, æੰÁY[¦∖ ŒÜÔPÙØŬ€FÏ
2	Deployment and retrieval of traffic control devices during roadway work	1	Stuck by vehicles	Face traffic and use spotter if not facing traffic, stay off the travelled roadway to extent practical, wear Class II (minimum) traffic vest. Familiarize yourself with work zone control layout prior to deploying devices.	
		2	Slips trips and falls on uneven road or land surfaces	Do not carry objects that obscure visibility of ground surface when walking, wear footgear with ankle support and good tread, use buddy system when carrying large bulky objects.	
		3	Lifting heavy or bulky signage or traffic channeling device	Brake down load to manageable size. Do not over reach to grab cones from the interior of the project vehicle. Use proper lifting techniques, maintain good vehicle housekeeping to easily retrieve control devices. Use buddy system to move heavy objects like barrels.	

2	Deployment and retrieval of traffic control devices during roadway work	4	Pinch points to hands on folding components of sign stands	Wear leather gloves or other suitable glove. Watch for hazard and avoid placing hands in pinch areas. Do not hurry through setup/take down task.	
3	Sharpen machete, brush axe or other cutting tool	1	Sharpening machete can cause lacerations and can generate metal shavings that can cause eye abrasions.	Secure blade to a sturdy fixture such as work bench and use vice. Make sure that sharp edge does not come in contact with fingers/body when sharpening. Sharpen blade 4"-10" above handle. Tip is not sharpened. Use Kevlar gloves and safety glasses.	
		2	Cuts from unsheathed/uncovered cutting tool upon completion of sharpening activity	Promptly sheath or cover cutting blade of cutting tool upon completion of sharpening task, do not "stick" machetes in ground until needed for use.	
4	Line cutting with machete	1	Improper use of the machete can cause lacerations	Do not reach or over-extend when cutting, and cut away from the body at 45 degree angle. Always keep machete sharpened. Do not use tool if the handle becomes wet/slippery. Never stick the blade into the groundsheath machete when not in use. See the Field H&S Handbook for detailed machete use instructions (section DD).	Field H&S Handbook Section D
		2	Utility lines can be accidentally severed during cutting	Inspect area for location of overhead lines prior to starting the task. Do not use machete when cutting vegetation that is close to utility lines. Use more appropriate tools such as garden clippers or shears.	Field H&S Handbook Section D
		3	Biologicals such as poisonous plants, bees/wasps, and other insects can be encountered during cutting of vegetation or brush.	Attempt to identify biological concerns prior to starting task. Use identification techniques outlined in the Field H&S Handbook.	Field H&S Handbook Section D
		4	Cardio and muscle fatigue can be experience from prolonged use of machete or when using machete for cutting of thick vegetation.	Take proper rest breaks, and rotate work jobs with co-workers. For thick vegetation, make sure the machete is the best tool for the job.	Field H&S Handbook Section D
		5	Impalement hazards from falls onto stumps of cut vegetation	Be aware of hazard and avoid walking in cut areas where vegetation exists that could present an impalement hazard. In areas where longer term work areas are cleared, take time to cut vegetation closer to ground surface without an angular cut.	Field H&S Handbook Section D
		6	Objects can fall once cut, or particles can become airborne getting into eyes or puncturing skin.	Wear hard hat, safety glasses and steel-toe shoes. Determine a safe fall zone. Do not use hard strokes when cutting with the machete to limit flying particles.	Field H&S Handbook Section D
		7	Fallen branches and vegetation can cause tripping hazard	Remove freshly cut limbs and brush from the work area to ensure balance, reduce slips and falls, and reduce obstructions.	Field H&S Handbook Section D
5	Line cutting using brush axe or chainsaw (must be approved by Party Chief).	1	Improper use of the bush axe or chainsaw can cause serious injury	Inspect equipment before use, and keep chain sharp. Hold the chainsaw with both hands, never cut above shoulder height. Keep saw close to your body. Carry brush axes sheathed and blade facing away from body. Do not carry brush axes when carrying other large or bulky objects.	Site clearing JLA

cha	ne cutting using brush axe or ainsaw (must be approved by arty Chief).	2	Struck by brush axe	Maintain proper separation distance when cutting, ensure anti-slip tape or other material on handles of brush axe to prevent slipping out of hands, wear gloves with good gripping capability.	Site clearing JLA
		3	Utility lines can be accidentally severed during cutting	Inspect area for location of overhead lines prior to starting the task. Note direction of fall for trees and ensure contact with utility lines will not occur	Site clearing JLA
		4	Objects can fall once cut, or particles can become airborne getting into eyes or puncturing skin.	Wear hard hat, safety glasses and steel-toe shoes. Determine a safe fall zone. to limit flying particles.	Site clearing JLA
		5	Fallen branches and vegetation can cause tripping hazard	Remove freshly cut limbs and brush from the work area to ensure balance, reduce slips and falls, and reduce obstructions.	Site clearing JLA
		6	Noise hazards (chainsaw)	Wear hearing protection (ear plugs or ear muffs)	Site clearing JLA
6 Re	emoval of manhole covers	1	Pinch points and scrape hazards when removing MH cover.	Do not place fingers under lid during removal, use shovels, pry bars, etc to place under lid edge to lift. Wear sturdy work glove. Wear steel toe boot, do not purposely drop lids.	
		2	Back/neck/arm/shoulder strains and hand blisters could occur from over lifting, or not lifting properly.	Use proper lifting techniques, keep back straight, lift with legs, use "J" Hook or pry bar, Buddy System required	
	uipment set-up, calibration d survey of target area	1	Slips/trips/falls can occur from walking on uneven ground surface.	Watch for uneven ground, debris, and trip hazards. If possible clear area of trip hazards. Wear gloves and heavy denim work pants to avoid cuts when working in heavy brush/briers. Use buddy system to spot for uneven ground while surveying.	
8 Pla	acement of stakes	1	Hands/fingers/arms can get struck by hammer/mallet. Splinters and lacerations can occur if stake splints during hammering.	Wear leather work gloves and safety glasses when placing stakes.	
9 Pla	acement of monuments	1	Back strain from digging holes or mixing concrete	Use proper shoveling techniques and keep back straight, Use right tool for the job.	refer to Concrete work JLA
		2	Exposure to concrete can cause skin irritation or illness	Wear impermeable glove during mixing and concrete placement, promptly wash exposed skin. Do not use bare hands to mix, place, or finish concrete.	refer to Concrete work JLA
		3	Inhalation of concrete dust during mixing	Keep face away from concrete when poured out of bag, Promptly wet concrete to be mixed.	refer to Concrete work JLA

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	work gloves (specify type)	Kevlar for machete use, leather for cutting	Required
Head Protection	hard hat		Required
Miscellaneous PPE	other	chainsaw chaps	Required

Туре	Supply	Description	Required
Miscellaneous	fire extinguisher		Required
Miscellaneous	first aid kit		Required
Miscellaneous	Other	snake chaps depending on work location	Recommended
Personal	water/fluid replacement		Required
Traffic Control	traffic cones	for roadway surveying	Required

### General

<u>v</u>	
Client Name	ARCADIS-AGMI
JSA ID	49
Job Name	Environmental-Soil sampling/well installation - manual
Task Description	hand augering
Project Number	00000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status	(3) Completed
Creation Date	2/6/2009 12:10:48 PM
Auto Closed	False

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Coppola, Mija	2/8/2009	2/6/2009		Coates, Gary	True
Developer (Primary Contact)	Coppola, Mija	2/9/2009	2/6/2009		Coates, Gary	True
HASP Reviewer	Coppola, Mija	2/8/2009	2/6/2009	True	Coates, Gary	True
Quality Reviewer	Vollertsen, Patricia	3/3/2009	3/3/2009		Coppola, Mija	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	02/06/2009	
Quality Reviewer	Vollertsen, Patricia	Quality	03/03/2009	$\begin{split} \tilde{S}[[ \cdot \hat{A} \alpha a a]^{A} [[ a A b a a A c - A b a A A A A A a A A A A A A A A A A A$

Job Step	Job Step Description	Potential Hazard	Critical Action	HSP Reference
1	Sampling set-up	1 Underground utilities could be encountered during hand augering	Follow the utility location policy and procedure.	Utility Location Policy ARCHSF019
		2 Muscle strains can occur from lifting heavy equipment in and out of vehicle	Park as close as possible to the sampling locations. Use lifting techniques as outlined in the Field H&S Handbook.	Utility Location Policy ARCHSF019
		3 slips/trips/falls could occur from uneven walking and working surfaces	Remove any gravel or debris from sample location. Gravel will get stuck in auger or will continue to fall back down in hole. A five gallon bucket with the bottom cut out will retain gravel from falling back down in the hole.	Utility Location Policy ARCHSF019
2	Installation of hand auger boring	1 Muscle Strains from pulling/pushing could occur when installing the boring, and when removing the auger from the hole	Stretch out Arms/Back/Shoulder Muscles prior to beginning. Using firm grip on handle, slowly turn auger and progress downward in 6" increments.Slowly pull auger from hole, use legs to pull auger out of hole. If water is encountered, a suction will be created when trying to remove the auger. Ask for assistance from another worker if you can't remove safely on your own.	
		2 Hand strain and blisters could develop from prolonged hand augering	Select proper gloves for task, usually leather type work gloves or mechanics style gloves. If hot spots develop on hands (Hot Spots are where blisters start to form) readjust gloves or change to better padded glove. If blisters begin to form, stop work so as not to worsen blistering.	
		3 Over-exertion could occur when trying to force an auger forward if there is refusal.	If refusal occurs, Stop Work. Remove Auger from hole and check hole with flashlight if possible. DO NOT overexert by using excessive force	
		4 Fatigue can occur due to strenuous nature of hand augering activities	Take rest breaks as needed or switch out task with another employee.	
3	Collect Sample Soil Sample	1 Staff can come into contact with impacted soils	Wear chemical protective gloves as outlined in the HASP, and wear safety glasses.	
		2 Sharp edges and broken glassware can cause lacerations	Discard any broken sample containers or glass. Do not overtighten sample containers.	
		3 Containerizing and moving soil cuttings can cause muscle strains	Dispose of left over soil cuttings in a drum or bucket and dispose properly. Only fill buckets half full due to weight and strength of bucket. Wear leather work gloves and use good lifting techniques when handling buckets.	
4	Decon Hand Auger	1 Exposure to COCs while deconing equipment.	Wear chemical protective gloves as outlined in the HASP, and wear safety glasses.	
		2 Cleaning solutions can splash while deconing equipment	Use PPE as outlined in the HASP, and try to minimize splashing.	
		3 The end of the hand auger has sharp edges, and lacerations can occur	Use brush to scrub off soils and not hands. Do not reach into the nose (the end with teeth) of the auger with hand.	
5	Fill in Sample Location	1 Open boreholes are a trip hazard	Fill in hole with sand or bentonite. Pack down chips as best as possible. Add a bit of DI Water to make chips swell and fill hole completely.	

5	Fill in Sample Location	Muscle strain can occur from lifting bags of sand and/or bentonite.	Use proper lifting techniques as detailed in the Field H&S handbook	
6	Installation of Temporary Piezometer/Well	Excessive noise can occur form driving well casing with hammer or macrocore sampler.	Wear hearing protection device.	see Concrete work JLA
		 Body parts or onlookers can be struck when using hand tools	Check swing radius above and around before driving well casing in bore hole. Wear leather gloves and safety glasses.	see Concrete work JLA
		Muscle strains can occur when pulling/pushing well materials into place	Stretch out Arms/Back/Shoulder Muscles prior to beginning. Take frequent breaks as fatigue sets in and if muscle cramps occur.	see Concrete work JLA
		Edges of PVC well casings have sharp edges especially when they have been cut, which can cause lacerations.	File the tops of well casings after they have been cut. Wear leather work gloves.	see Concrete work JLA
		Debris in the eyes can occur when working with soil, grout, and bentonite	Use disposable dust mask if excessive dust is created.	see Concrete work JLA

Туре	Personal Protective Equipment	Description	Required
Eye Protection	safety glasses		Required
Foot Protection	steel-toe boots		Required
Hand Protection	chemical resistant gloves (specify type)		Required
Hand Protection	work gloves (specify type)		Required
Head Protection	hard hat		Required
Hearing Protection	ear plugs		Required
Miscellaneous PPE	traffic vestClass II or III		Required
Respiratory Protection	dust mask		Recommended

Туре	Supply	Description	Required
Decontamination	Decon supplies (specify type)		Required
Miscellaneous	first aid kit		Required
Personal	eye wash (specify type)	bottle	Required
Traffic Control	traffic cones		Required

### General

<u>k</u>	
Client Name	ARCADIS-AGMI
JSA ID	2776
Job Name	Environmental-Drum sampling/handling
Task Description	Drum handling and sampling
Project Number	000000100000
Project Name	GENERAL OVERHEAD
PIC Name	
Project Manager	NO PROJECT MANAGER
Status	(3) Completed
Creation Date	6/10/2010 12:36:00 PM
Auto Closed	False

### User Roles

Role	Employee	Due Date	Completed	Approve	Supervisor	Active Employee
Created By	Byers, Susan	6/10/2010	6/10/2010		Vollertsen, Patricia	True
Developer (Primary Contact)	Moyers, Samuel	6/10/2010	6/10/2010		Coppola, Mija	True
HASP Reviewer	Coppola, Mija	6/24/2010	6/17/2010	True	Coates, Gary	True
Quality Reviewer	Mayo, Jessica	6/25/2010	6/25/2010		Bryz-Gornia, Jennifer	True

### **Reviewer Comments**

Role	Employee	Approval Status	Completed Date	Comments
HASP Reviewer	Coppola, Mija	Approve	06/17/2010	
Quality Reviewer	Mayo, Jessica	Quality	06/25/2010	Very thorough - Looks good!

Job Step	Job Step Description		Potential Hazard	Critical Action	HSP Reference
1	Q•]^&c/ÄC `{•Á{¦Áâ}+Á <sub>t</sub> ~ Ô` *ā}*ÊÁS^æàā}*Ê Ô¦^•cæ‡+ÊÁV^{]^¦æcč¦^ÊÁæa)å Uå[¦		Exposure to chemicals stored in drum or container.	Read drum labels for information about contents. Review all relevant MSDSs about chemical contents. If labels are not attached, call PM or Local H&S Representative.	None
		2	Contents of the drum can cause fire/explosion hazard.	Use air monitoring meters to screen drums. % LEL and VOCs (PPM). If either of the values are above the action levels described in the HASP or MSDS then Stop Work, move away from the area, and reassess the situation. Call PM and H&S staff for support.	None
2	Remove lids or bungs from Drums	1	Hand Injuries can occur from sharp edges, pinch points, and from use of hand tools.	Wear appropriate work gloves. When removing ring from drum, fingers can get pinched between ring and drum. Keep fingers clear of this space. Select proper tool for task. If large amount of drums will be encountered, use a speed or drum wrench.	Employee H&S Field book, Section III Subpart II, page 104. Also Section III Subpart L, page 38.

2	Remove lids or bungs from Drums	2	Rapid depressurization from empty or partially full drums can cause flying parts or volatile COCs releasing on staff.	Do not handle or open bulging drums (contact Corp H&S for assistance). Bleed any built up pressure by carefully loosening bung prior to removing ring. Keep face and arms away from bung opening when loosening. Slightly lift lid, insert end of air monitoring device to monitor air inside drum.	Employee H&S Field book, Section III Subpart II, page 104. Also Section III Subpart L, page 38.
		3	Use of mechanical tools to remove bolts from drum lids causes excessive noise.	Wear hearing protection.	Employee H&S Field book, Section III Subpart II, page 104. Also Section III Subpart L, page 38.
		4	Splashing can occur if filling drum, or collecting samples.	Wear eye and face protection. Pour liquids into drum slowly to minimize splashing.	Employee H&S Field book, Section III Subpart II, page 104. Also Section III Subpart L, page 38.
			When working with COCs that have fire/explosive properties, sparking or heat could cause fire/explosion.	Use brass or non Spark Hand Tools if such a hazard exists or is suspected.	Employee H&S Field book, Section III Subpart II, page 104. Also Section III Subpart L, page 38.
3	Sample Contents from Drums	1	Exposure to COCs can occur by contacting impacted contents.	Select proper dermal protection for task, at a minimum nitrile gloves should be worn. Wear appropriate eye face and body protection as outlined in the HASP.	
		2	Staff can be exposed to chemical vapors/fumes when sampling.	Conduct air monitoring as outlined in the HASP, and if required, select appropriate respiratory protection for the task.	
		3	Sharp edges and broken sample containers can cause lacerations.	Discard any broken sample ware or glass properly. Do not over tighten sample containers.	
		4	Chemical burns or skin irritation can occur from contact with sample preservatives.	Wear chemical protective gloves when collecting samples, or when handling damaged sample containers.	
4	Replace drum lids	1	Hand Injuries can occur from sharp edges, pinch points, and from use of hand tools.	see step 2 above	
5	Moving and Storing Drums	1	Drum storage areas can be accessed by the general public, or may not be secure.	Calculate how many drums will be stored in new location. Ensure that drums are not easily accessed by the general public. Do not store such that drums impede pedestrian or vehicular traffic.	
		2	Muscle strain can occur when lifting/pulling/pushing drums.	Drums that are full can weigh as much as 800 lbs. Use a lift assist device whenever possible, and use a team lift approach. When moving soil drum generated by drilling, have drillers use their equipment to move the drums. Using dolly, slightly lift drum away from dolly to install forks under drum. Slowly let drum come back down and rest on dolly. Using hook on top of dolly, ensure it latches on top of drum bung.	
		3	Body parts can be pinched between lift device, or drum and the ground.	Be aware of hand and foot placement during drum staging. Do not hurry through task.	
		4	When moving, the drum can tip or the dolly could become unstable from uneven ground surface.	Y @}Å, [çā] * Éxố@ Ási`{ Ásæ) Ási Á; c@ Ási[  ^ÁSi[` áÅs^8[{^Á} • cæà ^ +[{ Å}^^) ^A; [`]áÅs ^ [{^A\$; • cæà ^ +[{ Å}^^, ^A; Å; [] Åás ^ [] dæş^ Å[`c*Å äc@ Åsi`{ Å; [ā]; Ási { [çā] * ĒXY äc@ Åsi`{ Å, [ă]; Ási { [çā] * ĒXY äc@ Åsi`{ Å^8 `; ^Å; Åsi[  ^Ê @æş~Å; }^Å; ] [[^^^Å; ]] [Åsæ Å; Å; à[   ^ Ēxba) åÅ; c@ !Å{ ] [[^^^Å], æà Åsi[  ^Ê Pæş^Å ^ & Si } Åsi`{ Åsi; asà Åsi[  ^Ê Pæş ÅA & [; \^], Åsi & Åsi & Åsö & Åsa] [cc* ] + [Å tæ asæ Åz], ^å^ • dæg • Ēkba) Åkba) Åsi ] @æ æà å • Ása] [ * Ás@ Å; æÈ	

Туре	Personal Protective Equipment	Description	Required
Dermal Protection	chemical protective suit (specify type)		Required
Eye Protection	faceshield		Required
Eye Protection	safety goggles		Required
Hand Protection	chemical resistant gloves (specify type)	Nitrile	Required
Hand Protection	work gloves (specify type)		Required
Hearing Protection	ear plugs		Required

Туре	Supply	Description	Required
Miscellaneous	Other	dolly	Required

# **ARCADIS**

Appendix D

PPE Checklist



### PPE CHECKLIST

 $\mathbf{R}$  = Equipment required to be present on the site.  $\mathbf{O}$  = Optional equipment. Subcontractors must have the same equipment listed here as a minimum.

Description	I	_evel Of Protection	
(Put Specific Material or Type in Box)	D	C	В
Body			
Coveralls	0	NA	
Chemical Protective Suit	NA	R	
Splash Apron	0	NA	
Rain Suit	0	NA	
Traffic Safety Vest (reflective)	R	R	
Head	-		
Hard Hat (if does not create other hazard)	R	R	
Head Warmer (depends on temperature and weather conditions)	0	0	
Eyes & Face		1	
Safety Glasses (incorporate sun protection as necessary)	R	NA	
Goggles (based on hazard)	0	NA	
Splash Guard (based on hazard)	0	NA	
Ears			
Ear Plugs	R	R	
Ear Muffs	0	0	
Hands and Arms			
Outer Chemical Resistant Gloves	R (Nitrile)	R (Nitrile)	
Inner Chemical Resistant Gloves	NA	R (Nitrile)	
Insulated Gloves (depends on temperature)	0	0	
Work Gloves	0	0	
Foot			
Safety Boots (steel toe and shank)	R	R	
Rubber, Chemical Resistant Boots	NA	R	
Rubber Boots	0	0	
Disposable Boot Covers	0	0	
Respiratory Protection			
1/2 Mask APR	NA	NA	
Full Face APR	NA	R	
Dust Protection	0	NA	
Powered APR	NA	NA	
SCBA	NA	NA	
Air Line	NA	NA	

# **ARCADIS**

Appendix E

Forms

### **Employee Signature Form**

I certify that I have read, understand, and will abide by the safety requirements outlined in this HASP.

Printed Name	Signature	Date



Document Control Number:TGM - \_

TGM + project number plus date as follows: xxxxxxx.xxxx.xxxx - dd/mm/year

TAILGATE HEALTH & SAFETY MEETING FORM							
	•	•		•	Personnel who perform work oper heir attendance, at least daily.	ations on-	
Project Name:		1 410 1094		Project Lo			
Date:	Time:	Conducted b	эу:	Signature/	Signature/Title:		
Client:	<u> </u>	Client Conta	ict:	Subcontra	ctor companies:		
TRACKing	the Tailga	ate Meeti	ng				
<b>Think</b> through the	e Tasks (list the	tasks for the c	day):				
1			3		5		
2			4		6		
		y activities that	ox if there are any other AF t may pose hazards to ARC				
How will they	y be controlled?						
	pletion of a chec		e conducted that require pe r before work begins: Working at Height Excavation/Trenching	ermit <u>Doc #</u>	Confined Space	<u>Doc #</u>	
Mechanical Lif	ting Ops	[	Overhead & Buried Utili	ties	Other permit		
Discuss foll	owing questio	<b>NS</b> (for some revie	ew previous day's post activities). C	heck if yes :	Topics from Corp H&S to cove	ər?	
Incidents from	day before to re	eview?	Lessons learned from th	ne day before?	Any Stop Work Interventions	yesterday?	
Any corrective	e actions from ye	esterday?	Will any work deviate fr	rom plan?	If deviations, notify PM & clier	ıt	
JLAs or proce	dures are availa	able?	Field teams to "dirty" JL	As, as needed?	All equipment checked & OK?	,	
Staff has appro	opriate PPE?	Ī	Staff knows Emergency	Plan (EAP)?	Staff knows gathering points?		
Comments:	:						
<b>Recognize</b> the hazards (check all those that are discussed) (Examples are provided) and <b>Assess</b> the Risks (Low, Medium, High - circle risk level) - Provide an overall assessment of hazards to be encountered today and briefly list them under the hazard category.							
	lder, scaffold, trips)	(L M H)	Motion (i.e., traffic, moving w		Mechanical (i.e., augers, motors)	(L M H)	
Electrical (i.e., u	utilities, lightning)	(L M H)	Pressure (i.e., gas cylinders	, wells) (LMH)	Environment (i.e., heat, cold, ice)	(L M H)	
Chemical (i.e., f	uel, acid, paint)	(L M H)	Biological (i.e., ticks, poison	ivy) (LMH)	Radiation (i.e., alpha, sun, laser)	(L M H)	
Sound (i.e., mac	chinery, generators)	(L M H)	Personal (i.e. alone, night, n	not fit) (LMH)	Driving (i.e. car, ATV, boat, dozer)	(L M H)	
Continue	TRACK	Process	s on Page 2				

TAILGATE	HEALTH &	SAFETY MEETING F	OR	M - Pg. 2		
<b>C</b> ontrol the hazards (Check all and discuss the HASP, applicable JLAs, and other control proc					the day): Rev	view the
STOP WORK AUTHORITY (Must be addr Elimination Engineering controls General PPE Usage Personal Hygiene Emergency Action Plan (EAP) JLA to be developed/used (specify)	Substitutio Administra Hearing Co Exposure C Fall Protec	n tive controls onservation Guidelines		s below) Isolation Monitoring Respiratory Pl Decon Procec Work Zones/S Traffic Control Other <u>(specif</u>	lures Site Control	
Signature an	nd Certificat	tion Section - Site Sta	ff a	and Visitors	3	
Name/Company/Signature			Initial & Sign in Time	Initial & Sign out Time	I have read and understand the HASP	
Important Information and Numbers All site staff should arrive fit for work. If not, they should report to the supervisor any restrictions or concerns.	Visitor Name	e/Co - not involved in work		I will STOP the job a uncertain about heal hazard or additional project, job or task ha	th & safety or if anyon mitigation not record	one identifies a
In the event of an injury, employees will call WorkCare at 1.800.455.6155 and then notify the field supervisor who will, in turn, notify Corp H&S at 1.720.344.3844.	In	Out	1	I will be alert to any the work site or haza hazard assessments	irds not covered by	
In the event of a motor vehicle accident, employees will notify the field supervisor who will then notify Corp H&S at 1.720.344.3844 and then Corp Legal at 1.720.344.3756.	In	Out		If it is necessary to <b>S</b> TRACK; and then ar HASP as needed.		
In the event of a utility strike or other damage to property of a client or 3rd party, employees will immediately notify the field supervisor, who will then immediately notify Corp Legal at 1.678.373.9556 and Corp H&S at	In In	Out	-	I will not assist a su work unless it is abso I have done TRACK hazard.	olutely necessary ar	nd then only afte
1.720.344.3500				nazalu.		
Post Daily Activities Review - Re	eview at end of c	lay or before next day's work (	Che	eck those appl	icable and exp	olain:)
Lessons learned and best practices learn	ed today:					
Incidents that occurred today:						
Any Stop Work interventions today?						
Corrective/Preventive Actions needed for	future work:					
Any other H&S issues:						
<u>K</u> eep H&S 1 <sup>s</sup>	<sup>it</sup> in all th	ings		WorkCare - 1.800 Near Loss Hotlin		04

# ARCADIS

### **Real Time Air Monitoring Data Collection Form**

Document all air monitoring conducted on the Site below based on Section 7 of the HASP. Keep this form with the project files.

Site Name:			
Instrument:	Model:	Serial #:	

Calibration Method:	
(material used, settings, etc.)	
Calibration Results:	
Calibrated By:	

Activity Being Monitored	Compounds Monitored	Time	Reading	Action Required? Y/N

Describe Any Actions Taken as a Result of this Air Monitoring and Why:

## Visitor Acknowledgement and Acceptance of HASP Signature Form

By signing below, I waive, release and discharge the owner of the site and ARCADIS and their employees from any future claims for bodily and personal injuries which may result from my presence at, entering, or leaving the site and in any way arising from or related to any and all known and unknown conditions on the site.

Name	Company	Reason for Visit	Date/Time On Site	Date/Time Off Site

## Hazardous Materials Transportation Form

	Vehicle (place X in box)	Type (pick-up, car, box truck, etc.)
Personal		
Rental		
ARCADIS owned/leased		
Government owned		
Trailer		
Materials Transported	Quantity	Storage/Transport Container

List Trained Drivers:

## Hazardous Materials Shipment Form

Material Description and Proper Shipping Name (per DOT or IATA)	Shipment Quantity	DOT Hazard Classification	Shipment Method (air/ground)

List Shipper (i.e., who we are offering the shipment to):

List Trained Employee(s):

## **ARCADIS**

Appendix F

MSDSs

## Section 1. Chemical Product and Company Identification

Catalog Number(s)

00606-10, 00653-15, 00653-16, 00653-18, 00653-20, 00653-23, 00653-27, 00653-32, 00653-47, 00653-50, 00653-89, 35653-09, 35653-10, 35653-11, 35653-12, 35653-13, 35656-18, 35656-47 Product Identity

CONDUCTIVITY STANDARD SOLUTIONS, < 90 mS

Manufacturer's Name	Emergency Telepho	ne Number (24 hr)		
RICCA CHEMICAL COMPANY	CHEMTREC®:	800-424-9300		
Address (Number, Street, City, State, and ZIP Code)	Telephone Number	For Information		
P.O. Box 13090	817-461-5601			
	Date Prepared			
Arlington, Texas 76094	3-17-2000			
Section 2. Composition / Information on Ingredier	nts			
		Percent	Exposur	e Limits
Component	CAS Registry #	Concentration	ACGIH TLV	OSHA PEL
Potassium Chloride	7447-40-7	< 6	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

#### Section 3. Hazards Identification

## EMERGENCY OVERVIEW

Clear, colorless liquid. Non-flammable, non-toxic, non-corrosive. Does not present any significant health hazards.

TARGET ORGANS: eyes, skin.

EYE CONTACT: May cause irritation.

**INHALATION:** Not likely to be hazardous by inhalation.

SKIN CONTACT: May cause slight irritation.

**INGESTION:** Large doses may cause stomach upset.

#### CHRONIC EFFECTS / CARCINOGENICITY:

IARC – No NTP – No OSHA – No

#### TERATOLOGY (BIRTH DEFECT) INFORMATION:

Mutation data cited in 'Registry of Toxic Effects of Chemical Substances' for Potassium Chloride.

#### **REPRODUCTION INFORMATION:**

No information found in "Registry of Toxic Effects of Chemical Substances" or other information sources.

#### Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes.

PRODUCT IDENTITY: CONDUCTIVITY STANDARD SOLNS, < 90 mS CAT	NO (S): 00606-10, 00653-15, 00653-16, 00653-18, 00653-20, 00653-23, 00653-27, 00653-32, 00653-47, 006	853-50, 00653-
89, 35653-09, 35653-10, 35653-11, 35653-12, 35653-13, 35656-18, 35656-4	7	
EFFECTIVE DATE: 3-20-2006	MSDS NUMBER 00514 Rev 4	Page 1 of 3

# **ON®** MATERIAL SAFETY DATA SHEET

INHALATION: Remove to fresh air. Give artificial respiration if necessary.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes.

**INGESTION:** Dilute with water or milk. Call a physician if necessary.

FLAMMABLE PROPE	RTIES:		
FLASH POINT:	N/A	METHOD USED:	N/A
FLAMMABLE LIMITS			
LFL:	N/A	UFL:	N/A

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

#### Section 6. Accidental Release Measures

Absorb with suitable material (paper towels, etc.) and dispose of in accordance with local regulations. Small amounts may be flushed to the sewer with plenty of water.

#### Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

#### Section 8. Exposure Controls / Personal Protection

**ENGINEERING CONTROLS:** No specific controls are needed. Normal room ventilation is adequate.

**RESPIRATORY PROTECTION:** Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves are recommended.

EYE PROTECTION: Safety glasses or goggles.

## Section 9. Physical and chemical Properties

APPEARANCE:	Clear, colorless liquid	pH:	approximately 7
ODOR:	Odorless	BOILING POINT ( <sup>O</sup> C):	approximately 100
SOLUBILITY IN WATER:	Infinite	MELTING POINT ( <sup>0</sup> C):	approximately 0
SPECIFIC GRAVITY:	approximately 1.0 – 1.04	VAPOR PRESSURE:	N/A

#### Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

**INCOMPATIBILITY:** Bromine Trifluoride, Potassium Permanganate plus Sulfuric Acid.

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

## Section 11. Toxicological Information

LD<sub>50</sub>, Oral, Rat: 2600 mg/kg (Potassium Chloride), details of toxic effects not reported other than lethal dose value. Irritation: eye, rabbit (500mg/24 hr mild).

#### Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

#### Section 13. Disposal Considerations

Dilute with water and flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

#### Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME: Not regulated D.O.T. HAZARD CLASS: None U.N. / N.A. NUMBER: None PACKING GROUP: None D.O.T. LABEL: None

#### Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

**OSHA STATUS:** The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory. CERCLA REPORTABLE QUANTITY: Not reportable SARA TITLE III: SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No

SECTION 311/312 HAZARDOUS CATEGORIES: No SECTION 313 TOXIC CHEMICALS: No RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other	Information			
NFPA® Ratings: HMIS® Ratings:	Health: 0 Health: 0	Flammability: 0 Flammability: 0	,	Special Notice Key: None Protective Equipment: B (Protective eyewear, gloves)

Rev 1, 10-16-2000: (Section 1) added catalog numbers 35653-10, 35653-11, 35653-12, and 35653-13.

Rev 2, 12-06-2001: (Section 1) added catalog number 35653-09; revised description from 23µ - 80 mS.

Rev 3, 03-25-2003: Reviewed and approved, (Section 3) added mutation statement, (Section 11) added irritation data.

Rev 4, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

PRODUCT IDENTITY: CONDUCTIVITY STANDARD SOLNS, < 90 mS CAT NO (S): 00606-10, 00653-15, 00653-16, 00653-18, 00653-20, 00653-27, 00653-27, 00653-32, 00653-47, 00653-50, 00653-89, 35653-10, 35653-11, 35653-12, 35653-13, 35656-18, 35656-47 EFFECTIVE DATE: 3-20-2006 MSDS NUMBER 00514 Rev 4 Page 3 of 3

	RIAL SAF	ETY D	ATA SH	IEET
Section 1. Chemical Product and Company Identi	fication			
Catalog Number(s)				
00654-00, 05942-21, 05942-22, 05942-24, 05942-25	, 05942-26, 05942-2	7, 35653-01, 356	54-00	
Product Identity				
BUFFER, Standard, pH 4.01; BUFFER, High Accurac				
Manufacturer's Name	Emergency Telepho	ne Number (24 hr)		
RICCA CHEMICAL COMPANY	CHEMTREC®:	800-424-9300		
Address (Number, Street, City, State, and ZIP Code)	Telephone Number	For Information		
P.O. Box 13090	817-461-5601			
	Date Prepared			
Arlington, Texas 76094	3-7-2000			
Section 2. Composition / Information on Ingredier	nts			
		Percent	Exposur	e Limits
Component	CAS Registry #	Concentration	ACGIH TLV	OSHA PEL
Potassium Acid Phthalate	877-24-7	0.95 - 1.05	N/A	N/A
Preservative*	proprietary	<0.5	N/A	N/A
*(No Mercury compounds or Formaldehyde) Inert Dye	proprietary	<0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

#### Section 3. Hazards Identification

Non-flammable, non-toxic, non-corrosive. Does not present any significant health hazards. Wash areas of contact with water.

#### 

TARGET ORGANS: eyes, skin.

**EYE CONTACT:** May cause slight irritation.

**INHALATION:** Not likely to be hazardous by inhalation.

SKIN CONTACT: May cause slight irritation.

**INGESTION:** Large doses may cause nausea, vomiting, diarrhea and cramps.

CHRONIC EFFECTS / CARCINOGENICITY:

IARC – No NTP – No OSHA – No

#### TERATOLOGY (BIRTH DEFECT) INFORMATION:

No information found in "Registry of Toxic Effects of Chemical Substances" or other information sources.

#### **REPRODUCTION INFORMATION:**

No information found in "Registry of Toxic Effects of Chemical Substances" or other information sources.

 PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 4.01
 CATALOG NUMBER (S): 00654-00, 35654-00, 05942-21, 05942-22, 05942-24, 05942-26, 05942-27, 35653-01

 EFFECTIVE DATE: 03-20-2006
 MSDS NUMBER 00506 Rev 3
 Page 1 of 3

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## Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

**INGESTION:** Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fig		asures		
FLAMMABLE PROPE	RHES:			
FLASH POINT:	N/A	METHOD USED:	N/A	
FLAMMABLE LIMITS				
LFL:	N/A	UFL:	N/A	
EXTINGUISHING MEE	NA: Use ar	y means suitable for extinguishing surrounding	fire.	

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

#### Section 6. Accidental Release Measures

Absorb with suitable material and dispose of in accordance with local regulations.

#### Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

#### Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

**RESPIRATORY PROTECTION:** Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

#### Section 9. Physical and chemical Properties

APPEARANCE:	Clear, red colored liquid	pH:	4
ODOR:	odorless	BOILING POINT ( <sup>O</sup> C):	approximately 100
SOLUBILITY IN WATER:	infinite	MELTING POINT ( <sup>O</sup> C):	approximately 0
SPECIFIC GRAVITY:	approximately 1	VAPOR PRESSURE:	N/A

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

#### **INCOMPATIBILITY:** Nitric Acid

PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 4.01 CATALOG NUMBER (S): 00654-00, 35654-00, 05942-21, 05942-22, 05942-24, 05942-25, 05942-26, (MSDS NUMBER 00506 Rev 3	
EFFECTIVE DATE: 03-20-2006 MSDS NUMBER 00506 Rev 3	Page 2 of 3

## **OAKION®** MATERIAL SAFETY DATA SHEET

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Carbon and Potassium.

HAZARDOUS POLYMERIZATION: Will not occur.

#### Section 11. Toxicological Information

LD50, Oral, Rat: >3200 mg/kg (Potassium Acid Phthalate), details of toxic effects not reported other than lethal dose value.

#### Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

#### Section 13. Disposal Considerations

Dilute with water, neutralize with weak sodium hydroxide solution, and then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

Section 14. Transport Information (Not meant to be all inclusive)				
D.O.T. SHIPPING NAME:	Not regulated			
D.O.T. HAZARD CLASS:	None			
U.N. / N.A. NUMBER:	None			
PACKING GROUP:	None			
D.O.T. LABEL:	None			
Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)				

Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

OSHA STATUS: The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

TSCA STATUS: All components of this solution are listed on the TSCA Inventory. **CERCLA REPORTABLE QUANTITY:** Not reportable SARA TITLE III: SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: NO SECTION 311/312 HAZARDOUS CATEGORIES: NO

SECTION 313 TOXIC CHEMICALS: No

RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed

Section 16. Other Information					
NFPA® Ratings: HMIS® Ratings:	Health: 1 Health: 1	Flammability: 0 Flammability: 0	•	Special Notice Key: None Protective Equipment: B (Protective eyewear, gloves)	

Rev 1, 10-16-2000: (Section 1) added catalog number 35653-01.

Rev 2, 03-25-2003: Reviewed and approved.

Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 4.01 EFFECTIVE DATE: 03-20-2006 CATALOG NUMBER (S): 00654-00, 35654-00, 05942-21, 05942-22, 05942-24, 05942-25, 05942-26, 05942-27, 35653-01 MSDS NUMBER 00506 Rev 3

Section 1. Chemical Product and Company Identif	ication			
Catalog Number(s)				
00654-04, 35654-04, 05942-41, 05942-42, 05942-44,	05942-45, 35653-02	2		
Product Identity				
BUFFER, Standard, pH 7.00 (Color Coded Green)				
Manufacturer's Name	Emergency Telepho			
RICCA CHEMICAL COMPANY	CHEMTREC®: 8			
Address (Number, Street, City, State, and ZIP Code)	Telephone Number I	For Information		
P.O. Box 13090	817-461-5601			
	Date Prepared			
Arlington, Texas 76094	3-8-2000			
Section 2. Composition / Information on Ingredien	ts			
		Percent	Exposur	
Component	CAS Registry #	Concentration	ACGIH TLV	OSHA PEL
Sodium Phosphate, Dibasic	7558-79-4	< 1	N/A	N/A
Potassium Phosphate, Monobasic	7778-77-0	< 1	N/A	N/A
Preservative* *(No Mercury Compounds or Formaldehyde)	Proprietary	< 0.1	N/A	N/A
Inert Dye	Proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A
Section 3. Hazards Identification				

\*\*\*\*

EMERGENCY OVERVIEW

Non-flammable, non-toxic, non-corrosive. Does not present any significant health hazards. May cause irritation. Wash areas of contact with water

## 

TARGET ORGANS: eyes, skin.

EYE CONTACT: May cause slight irritation.

INHALATION: May cause allergic respiratory reaction to those allergic to phosphates.

**SKIN CONTACT:** May cause slight irritation to those allergic to phosphates.

INGESTION: Large doses may cause stomach upset.

CHRONIC EFFECTS / CARCINOGENICITY:

IARC – No NTP – No OSHA – No **TERATOLOGY (BIRTH DEFECT) INFORMATION:** 

No information found in "Registry of Toxic Effects of Chemical Substances" or other information sources.

#### **REPRODUCTION INFORMATION:**

No information found in "Registry of Toxic Effects of Chemical Substances" or other information sources.

PRODUCT IDENTITY: BUFFER, Standard, pH 7.00 (Color Coded Gre	en) CAT. NO (S): 00654-04, 05942-4	1, 35654-04, 05942-42, 05942-44, 05942-45, 35653-02
	MSDS NUMBER 00507 Rev 3	Page 1 of 3

#### Section 4. First Aid Measures - In all cases, seek qualified evaluation.

**EYE CONTACT:** Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

**INGESTION:** Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fighting Measures				
FLAMMABLE PROPE	RTIES:			
FLASH POINT:	N/A	METHOD USED:	N/A	
FLAMMABLE LIMITS				
LFL:	N/A	UFL:	N/A	
EXTINGUISHING MED	IA: Use any m	eans suitable for extinguishing surrounding	fire.	

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

#### **Section 6. Accidental Release Measures**

Absorb with suitable material (vermiculite, clay, etc.) and dispose of in accordance with local regulations. Check with local agencies for the proper disposal of phosphate containing solutions.

#### Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

#### Section 8. Exposure Controls / Personal Protection

**ENGINEERING CONTROLS:** No specific controls are needed. Normal room ventilation is adequate.

**RESPIRATORY PROTECTION:** Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

#### Section 9. Physical and chemical Properties APPEARANCE: pH: Clear, green liquid ODOR: BOILING POINT (°C): Odorless approximately 100 SOLUBILITY IN WATER: MELTING POINT (°C): Infinite approximately 0 VAPOR PRESSURE: SPECIFIC GRAVITY: N/A approximately 1

Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

**INCOMPATIBILITY:** None identified.

HAZARDOUS DECOMPOSITION PRODUCTS: Phosphorus oxides may form when heated to decomposition.

PRODUCT IDENTITY: BUFFER, Standard, pH 7.00 (Color Coded	Green) CAT. NO (S): 00654-04, 05942-41,	35654-04, 05942-42, 05942-44, 05942-45, 35653-02
EFFECTIVE DATE: 3-20-2006	MSDS NUMBER 00507 Rev 3	Page 2 of 3

HAZARDOUS POLYMERIZATION: Will not occur.

#### Section 11. Toxicological Information

LD50, Oral, Rat: (Sodium Phosphate Dibasic) 17 gm/kg; LD50, Dermal, Rabbit: (Potassium Phosphate Monobasic) >4640 mg/kg; details of toxic effects not reported other than lethal dose value.

#### Section 12. Ecological Information

**ECOTOXICOLOGICAL INFORMATION:** No information found.

CHEMICAL FATE INFORMATION: No information found.

#### Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow for the flushing of phosphate containing solutions. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

## Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME: Not regulated D.O.T. HAZARD CLASS: None U.N. / N.A. NUMBER: None PACKING GROUP: None D.O.T. LABEL: None

#### Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

**OSHA STATUS:** The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

**TSCA STATUS:** All components of this solution are listed on the TSCA Inventory or are mixtures (hydrates) of items listed on the TSCA Inventory.

**CERCLA REPORTABLE QUANTITY:** Sodium Phosphate, Dibasic - 5,000 pounds.

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No SECTION 311/312 HAZARDOUS CATEGORIES: No SECTION 313 TOXIC CHEMICALS: No RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

**PENNSYLVANIA**: Sodium Phosphate Dibasic is listed as an environmental hazard on the state Hazardous Substance list.

Section 16. Other	Information			
NFPA Ratings: HMIS® Ratings:	Health: 1 Health: 1	Flammability: 0 Flammability: 0	Reactivity: 0 Reactivity: 0	Special Notice Key: None Protective Equipment: B (Protective eyewear, gloves)

Rev 1, 8-25-2000: (Section 2) corrected concentration of preservative from 1 - 2 to < 0.1%. Rev 2, 03-25-2003: Reviewed and approved, (Section 15) added CERCLA reportable quantity.

Rev 3, 03-20-2006: Reviewed and approved, (Section

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

PRODUCT IDENTITY: BUFFER, Standard, pH 7.00 (Color Coded G	reen) CAT. NO (S): 00654-04, 05942-41, 356	54-04, 05942-42, 05942-44, 05942-45, 35653-02
EFFECTIVE DATE: 3-20-2006	MSDS NUMBER 00507 Rev 3	Page 3 of 3

Section 1. Chemical Product and Company Iden	tification			
Catalog Number(s)				
00654-08, 35654-08, 05942-61, 05942-62, 05942-6	64, 05942-65, 05942-66	6, 05942-67, 356	53-03	
Product Identity				
BUFFER, Standard, pH 10.00; BUFFER, High Accu				
Manufacturer's Name	Emergency Telepho	ne Number (24 hr)		
RICCA CHEMICAL COMPANY	CHEMTREC®:	800-424-9300		
Address (Number, Street, City, State, and ZIP Code)	Telephone Number	For Information		
P.O. Box 13090	817-461-5601			
	Date Prepared			
Arlington, Texas 76094	3-8-2000			
Section 2. Composition / Information on Ingredi	ents			
		Percent	Exposur	
Component	CAS Registry #	Concentration	ACGIH TLV	OSHA PEI
Sodium Carbonate	497-19-8	< 1	N/A	N/A
Sodium Bicarbonate	144-55-8	< 1	N/A	N/A
Preservative*	proprietary	< 0.1	N/A	N/A
*(No Mercury compounds or Formaldehyde) Inert Dye	proprietary	< 0.1	N/A	N/A
Water, Deionized	7732-18-5	Balance	N/A	N/A

## Section 3. Hazards Identification

\*\*\*\*

**EMERGENCY OVERVIEW** 

Non-flammable, non-toxic, non-corrosive. Does not present any significant health hazards. Wash areas of contact with water.

TARGET ORGANS: eyes, skin.

**EYE CONTACT:** May cause slight irritation.

INHALATION: Not likely to be hazardous by inhalation.

SKIN CONTACT: May cause slight irritation.

INGESTION: Large doses may cause nausea, vomiting, diarrhea and cramps.

CHRONIC EFFECTS / CARCINOGENICITY: IARC – No NTP – No OSHA – No

#### TERATOLOGY (BIRTH DEFECT) INFORMATION:

Mutation data cited in "Registry of Toxic Effects of Chemical Substances" for Sodium Bicarbonate in rats.

PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 10.00	CATALOG NUMBER (S): 00654-08, 35654-08, 05942-61, 05942-62, 05942-64,	05942-65, 05942-66, 05942-67, 35653-03
EFFECTIVE DATE: 3-20-2006	MSDS NUMBER 00509 Rev 3	Page 1 of 4

## MATERIAL SAFETY DATA SHEET

#### **REPRODUCTION INFORMATION:**

OAKION®

Reproductive data cited in "Registry of Toxic Effects of Chemical Substances" for Sodium Bicarbonate and Sodium Carbonate in mice.

#### Section 4. First Aid Measures – In all cases, seek qualified evaluation.

EYE CONTACT: Irrigate immediately with large quantity of water for at least 15 minutes. Call a physician if irritation develops.

INHALATION: Remove to fresh air. Give artificial respiration if necessary. If breathing is difficult, give oxygen.

SKIN CONTACT: Flush with plenty of water for at least 15 minutes. Call a physician if irritation develops.

**INGESTION:** Dilute with water or milk. Call a physician if necessary.

Section 5. Fire Fig	ghting	Measures		
FLAMMABLE PROPE	RTIES:			
FLASH POINT:	N/A	METHOD USED:	N/A	
FLAMMABLE LIMITS				
LFL:	N/A	UFL:	N/A	
EXTINGUISHING MED	IA: Use	any means suitable for extinguishing surrounding	fire.	

FIRE & EXPLOSION HAZARDS: Not considered to be a fire or explosion hazard.

FIRE FIGHTING INSTRUCTIONS: Use normal procedures/instructions.

FIRE FIGHTING EQUIPMENT: Use protective clothing and breathing equipment appropriate for the surrounding fire.

#### Section 6. Accidental Release Measures

Absorb with suitable material and treat as normal refuse. Small amounts of the liquid may be flushed to the drain with excess water. Always dispose of in accordance with local regulations.

### Section 7. Handling and Storage

As with all chemicals, wash hands thoroughly after handling. Avoid contact with eyes and skin. Protect from freezing and physical damage. SAFETY STORAGE CODE: GENERAL

#### Section 8. Exposure Controls / Personal Protection

ENGINEERING CONTROLS: No specific controls are needed. Normal room ventilation is adequate.

**RESPIRATORY PROTECTION:** Normal room ventilation is adequate.

SKIN PROTECTION: Chemical resistant gloves.

EYE PROTECTION: Safety glasses or goggles.

## Section 9. Physical and chemical Properties

APPEARANCE:	Clear, blue colored liquid	pH:	10
ODOR:	Odorless	BOILING POINT ( <sup>o</sup> C):	approximately 100
SOLUBILITY IN WATER:	Infinite	MELTING POINT ( <sup>O</sup> C):	approximately 0
SPECIFIC GRAVITY:	approximately 1	VAPOR PRESSURE:	N/A

PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 10.00	CATALOG NUMBER (S): 00654-08, 35654-08, 05942-61, 05942-62, 05942-64, 0	5942-65, 05942-66, 05942-67, 35653-03
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#### Section 10. Stability and Reactivity

CHEMICAL STABILITY: Stable under normal conditions of use and storage.

#### **INCOMPATIBILITY: Acids**

HAZARDOUS DECOMPOSITION PRODUCTS: Oxides of Sodium.

HAZARDOUS POLYMERIZATION: Will not occur.

#### Section 11. Toxicological Information

LD50, Oral, Rat: 4090 mg/kg (Sodium Carbonate), 4220 mg/kg (Sodium Bicarbonate), details of toxic effects not reported other than lethal dose value.

#### Section 12. Ecological Information

ECOTOXICOLOGICAL INFORMATION: No information found.

CHEMICAL FATE INFORMATION: No information found.

#### Section 13. Disposal Considerations

Dilute with water, then flush to sewer if local regulations allow. If not allowed, save for recovery or recycling in an approved waste disposal facility. Always dispose of in accordance with local, state and federal regulations.

#### Section 14. Transport Information (Not meant to be all inclusive)

D.O.T. SHIPPING NAME: Not regulated D.O.T. HAZARD CLASS: None U.N. / N.A. NUMBER: None PACKING GROUP: None D.O.T. LABEL: None

#### Section 15. Regulatory Information (Not meant to be all inclusive - selected regulation represented)

**OSHA STATUS:** The above items either do not contain any specifically hazardous material or the potentially hazardous material is present in such low concentration that the items do not present any immediate threat to health and safety. These items do not meet the OSHA Hazard Communication Standard (29 CFR 1910.1200) definition of a hazardous material.

**TSCA STATUS:** All components of this solution are listed on the TSCA Inventory.

**CERCLA REPORTABLE QUANTITY:** Not reportable

SARA TITLE III:

SECTION 302 EXTREMELY HAZARDOUS SUBSTANCES: No SECTION 311/312 HAZARDOUS CATEGORIES: No SECTION 313 TOXIC CHEMICALS: No RCRA STATUS: No

CALIFORNIA PROPOSITION 65: Not listed.

Section 16. Other	Information			
NFPA® Ratings:	Health: 1	Flammability: 0	Reactivity: 0	Special Notice Key: None
HMIS® Ratings:	Health: 1	Flammability: 0	Reactivity: 0	Protective Equipment: B
-				(Protective eyewear, gloves)

Rev 1, 01-15-2003: added catalog number 35653-03. Rev 2, 03-25-2003: Reviewed and approved. Rev 3, 03-20-2006: Reviewed and approved.

When handled properly by qualified personnel, the product described herein does not present a significant health or safety hazard. Alteration of its characteristics by concentration, evaporation, addition of other substances, or other means may present hazards not specifically addressed herein and

PRODUCT IDENTITY: BUFFER, Standard and High Accuracy, pH 10.00	CATALOG NUMBER (S): 00654-08, 35654-08, 05942-61, 05942-62, 05942-64, 05942-65, 05942-66	, 05942-67, 35653-03
EFFECTIVE DATE: 3-20-2006	MSDS NUMBER 00509 Rev 3	Page 3 of 4



## MATERIAL SAFETY DATA SHEET

which must be evaluated by the user. The information furnished herein is believed to be accurate and represents the best data currently available to us. No warranty, expressed or implied, is made and RICCA CHEMICAL COMPANY assumes no legal responsibility or liability whatsoever resulting from its use.

HANNA Instruments

## HI 93703-0 Primary Standard –0 FTU

## Health & Safety data sheet According to EC Directive 91/155/EC and following amendments

Date of issue: 03 January 2008.

#### SECTION 1 - IDENTIFICATION OF THE PRODUCT AND OF THE COMPANY Product name:

#### **Application:**

• HI 93703-0 Primary Standard – 0 FTU. Calibration Solution for turbidity measurements.

Manufacturer identification: Hanna Instruments Italia s.r.l. viale delle Industrie, 12/A 35010 Villafranca Padovana, Italy tel. n° .: +39-049-9070211

Emergency Telephone n.°: No hazardous product

#### SECTION 2 - COMPOSITION/INFORMATION ON INGREDIENTS Aqueous solution.

**SECTION 3 - HAZARD IDENTIFICATION** 

No hazardous product as specified in Directive 67/548/EEC.

#### **SECTION 4** - FIRST AID MEASURES

- After inhalation : NA
- After skin contact : NA
- : NA After eye contact
- : NA After swallowing

#### **SECTION 5 – FIRE-FIGHTING MEASURES**

- Suitable extinguishing media
- In adaptation to materials stored in the neighborhood.
- Special risks:
- None.
- · Additional information:
  - No special fire precautions are required.

#### **SECTION 6** - ACCIDENTAL RELEASE MEASURES

- **Personal precautions:**
- None
- **Environmental precautions:** 
  - None

#### **SECTION 7** - HANDLING AND STORAGE

Handling:

- No restrictions

#### Storage:

- Keep container closed and protected from direct sunlight.
- Store at room temperature (+15°C to +25°C).

#### **SECTION 8 - EXPOSURE CONTROL/PERSONAL PROTECTION**

- Personal protective equipment:
  - Not required.



HANNA Instruments

## HI 93703-0 Primary Standard –0 FTU

## Health & Safety data sheet

According to EC Directive 91/155/EC and following amendments

## **SECTION 9 - PHYSICAL/CHEMICAL PROPERTIES**

- **Appearance and odor** : clear liquid.
- Odor : odorless.
- Solubility in water : soluble :~0 °C
- Melting point
- :~ 100 °C Boiling point
- pH value at 20°C :~6.5

#### **SECTION 10 - STABILITY AND REACTIVITY**

- Conditions to be avoided:
  - Strong heating (above boiling point).
  - Stable in the recommended storage conditions.
- Hazardous decomposition products: - In the event of fire: see section 5.

#### **SECTION 11 - TOXICOLOGICAL INFORMATION**

No toxic effects are to be expected when the product is handled appropriately.

#### **SECTION 12 - ECOLOGICAL INFORMATION**

No environmental hazard.

#### **SECTION 13** - DISPOSAL CONSIDERATIONS

- Waste disposal:
  - Can be safely disposed off as an ordinary refuse.

#### **SECTION 14 - TRANSPORT INFORMATION**

Not subject to transport regulations.

#### **SECTION 15 - REGULATORY INFORMATION**

#### Labeling according to EC Directives:

Symbol: R-phrases: S-phrases: \_ Contains :

#### **SECTION 16 - OTHER INFORMATION**

- Supersedes edition of : June 2002
- Reason for revision : general update
- : NA Not Applicable Legend ND Not Determined

THE INFORMATION CONTAINED HEREIN IS BASED ON THE PRESENT STATE OF OUR KNOWLEDGE. IT CHARACTERIZES THE PRODUCT WITH REGARD TO THE APPROPRIATE SAFETY PRECAUTIONS. IT DOES NOT REPRESENT A GUARANTEE OF THE PROPERTIES OF THE PRODUCT.

- $: ~ 1 \text{ g/cm}^3$ density at 20°C
- flash point : NA explosive properties : NA
- explosion limits : NA
- ignition temperature : NA
- · Substances to be avoided: - None.
- Hazardous Polymerization:
  - Will not occur.



HANNA instruments

#### HI 93703-10 Primary Standard – 10 FTU

## Health & Safety data sheet According to EC Directive 91/155/EC

Date of issue: 03 January 2008

#### SECTION 1 - IDENTIFICATION OF THE PRODUCT AND OF THE COMPANY Product name:

• HI 93703-10 AMCO-AEPA Primary Standard – 10FTU Application:

Calibration Solution for turbidity measurements

Manufacturer identification: Hanna Instruments Italia s.r.l. viale delle Industrie, 12/A 35010 Villafranca Padovana, Italy tel. n°.:+39-049-9070211

**Emergency Telephone n.°:** No hazardous product

<u>SECTION 2</u> – COMPOSITION/INFORMATION ON INGREDIENTS Aqueous solution.

#### **SECTION 3** - HAZARD IDENTIFICATION

No hazardous product as specified in Directive 67/548/EEC.

#### **SECTION 4** - FIRST AID MEASURES

- After inhalation : NA
- After skin contact : NA
- After eye contact : NA
- After swallowing : NA

## **SECTION 5** – FIRE-FIGHTING MEASURES

- Suitable extinguishing media
- In adaptation to materials stored in the neighborhood.
- Special risks:

- Specific Hazard(s): emits toxic fumes under fire conditions. The following may develop in event of fire: carbon monoxide, carbon dioxide, alkylbenzene, vinylbenzene, naphthalene, benzaldehydes and phenol.

- Additional information:
  - No special fire precautions are required.

#### **SECTION 6** - ACCIDENTAL RELEASE MEASURES

- Personal precautions:
   None
- Environmental precautions:
   None

## SECTION 7 - HANDLING AND STORAGE

- Handling:
  - No restrictions

#### • Storage:

- Keep container closed and protected from direct sunlight.
- Store at room temperature (+15°C to +25°C).

#### **SECTION 8** - EXPOSURE CONTROL/PERSONAL PROTECTION

- Personal protective equipment:
  - Not required.

ETIC703-10



HANNA instruments

## HI 93703-10 Primary Standard – 10 FTU

## Health & Safety data sheet According to EC Directive 91/155/EC

## **SECTION 9 - PHYSICAL/CHEMICAL PROPERTIES**

- Appearance and odor : clear to opaque liquid. Odor : odorless.
- Solubility in water : soluble
- Melting point :~0 °C
- **Boiling point** :~ 100 °C
- pH value at 20°C :~6.5

#### **SECTION 10 - STABILITY AND REACTIVITY**

- Conditions to be avoided:
  - Strong heating (above boiling point).
  - Stable in the recommended storage conditions.
- Hazardous decomposition products: - In the event of fire: see section 5.

#### **SECTION 11 - TOXICOLOGICAL INFORMATION**

- In case of inhalation : NA.
- : irritant effects, danger of skin absorption. In case of skin contact
- In case of skin absorption : wash hands / use moisturizer if dryness develops.
- In case of eye contact : flush with water several times.
- In case of ingestion : not hazardous.
- Further data : no toxic effects are to be expected when the product is handled appropriately.

#### **SECTION 12** - ECOLOGICAL INFORMATION

No environmental hazard.

#### **SECTION 13 - DISPOSAL CONSIDERATIONS**

- Waste disposal:
  - Can be safely disposed off as an ordinary refuse.

#### **SECTION 14** - TRANSPORT INFORMATION

Not subject to transport regulations.

#### **SECTION 15 - REGULATORY INFORMATION**

#### Labeling according to EC Directives:

Symbol:	-
R-phrases :	-
S-phrases :	-
Contains :	-

#### **SECTION 16** - OTHER INFORMATION

- **Supersedes edition of** : / (1<sup>st</sup> edition)
- : NA Not Applicable Legend
  - ND Not Determined

THE INFORMATION CONTAINED HEREIN IS BASED ON THE PRESENT STATE OF OUR KNOWLEDGE. IT CHARACTERIZES THE PRODUCT WITH REGARD TO THE APPROPRIATE SAFETY PRECAUTIONS. IT DOES NOT REPRESENT A GUARANTEE OF THE PROPERTIES OF THE PRODUCT.

- $: ~ 1 \text{ g/cm}^3$ density at 20°C flash point : NA
- explosive properties : NA
- explosion limits : NA
- ignition temperature :NA
- Substances to be avoided: - Organic compounds.
- Hazardous Polymerization:
  - Will not occur.

ETIC703-10

# MATERIAL DATA SAFETY SHEET

HMIS Ratings Health: 0 Flammability: 0 Reactivity: 0

	HEEI Reactivity: 0						
	ine® solution -	Product #s 32-0	00400, 32-0004	01, 32-000502, 32	-001050, 32-001052	2	
Section I							
Manufacturer:	Fendall, Inc.		Emergency Telephone: 1-401-232-1200				
Address:         825 East Highway 151         Information Telephone: 800-543-4842							
Platteville, WI 53818 USA Date Prepared: 11/30/06					·		
Section II - Haza	ardous Ingredie	nts/Identify Info	rmation				
Hazardous Com			OSHA	ACGIH	Other limits	% (optional)	
(Specific Chemi	cal Identity; Co	mmon Name(s))	PEL	TLV	recommended		
BENZALKONIUM CHLORIDE CAS #8001-54-5			NONE	NONE	N/A	<0.1%	
Section III - Phy	sical/Chemical	Characteristics					
Boiling Point: 2	00°F (93.3°C)		Specific Grav	ity (H2O)=1: NOT	DETERMINED		
Vapor Pressure	(mm Hg.): 760		Melting Point	:: N/A			
Vapor Density (	Air = 1): NOT DE	TERMINED.	Evaporation	Rate (Butyl Acetat	e = 1): NOT DETER	RMINED	
Solubility in Wa	ter: 100%		<b>.</b>				
Appearance and	d Odor: COLOR	LESS LIQUID W	ITH NO DISCEI	RNABLE ODOR.			
Section IV - Fire	and Explosion	Hazard Data					
Flash Point (Me	thod Used): N/A	۸	Flammable L	imits:	LEL: N/A	UEL: N/A	
Extinguishing M	-		LE AQUEOUS	SOLUTION.			
Special Fire Fig							
Unusual Fire an							
Section V - Rea	-						
Stability	Unstab	le: NO	Conditions to	Avoid: THIS PRO	DUCT IS STABLE	AND CONSIDERED NON-REACTIVE	
otability	Stable:				OF STORAGE AN		
Incompatibility							
Hazardous Dec							
Hazardous	-	cur: NO		Avoid: NONE			
Polymerization	-	t Occur: YES					
Section VI - Hea			1				
Route(s) of Enti			O Indestion <sup>4</sup>	7 YES			
	-				20 LITERS MAY C	AUSE GASTRIC IRRITATION.	
Carcinogenicity		IARC Monogra		HA Regulated? N			
Signs and Sym							
Medical Conditi			NDOGURO: NI/A				
						GESTION OF LARGE VOLUMES	
					STRIC IRRITATION		
Section VII - Pre	,			FATILINT FOR GA			
		-				JTION IS NOT RCRA	
HAZARDOUS W		erial is Released	or Spilled: FL	USH AREA WITH	WATER. THE SOLU		
Waste Disposal							
•		adling and Ctari					
110°F (43°C) FO		-	ng: DO NOT F	REELE OR EAPOS	BETOTEWFERAT	JRES IN EXCESS OF	
Other Precautio		ERIODS.					
Section VIII - Co							
Respiratory Pro							
Ventilation		Exhaust: N/A			Special: N/A		
		nical: N/A			Other: N/A		
Protective Glov				Eye Protection:	N/A		
Other Protective							
Work Hygienic	Practices: N/A						
						Part No. 32-003731 Rev F	

# **Material Safety Data Sheet**

June 1, 1999

## YSI Incorporated 1725 Brannum Lane

Yellow Springs, OH 45387

USA

C-P# 05478-60

Information and Emergency Phone: (937) 767-7241

Page 1 of 2

## SECTION 1 - MATERIAL IDENTIFICATION

PRODUCT NAME: YSI 3682 Zobell Solution Chemical Type: \_\_\_\_\_Inorganic\_chloride / cyanide

CAS No. \_\_\_\_\_\_

## SECTION 2 - HAZARDOUS / IMPORTANT INGREDIENTS

<u>Chemical</u>	CAS No.	PERCENT	PEL/TLV	CARCINOGEN (OSHA, NTP, IARC)
Potassium chloride	7447-40-7	72 - 78%	none	no
Potassium ferrocyanide, trihydrate	14459-95-1	10 - 15%	none	no
Potassium ferricyanide	13746-66-2	10 - 15%	none	no

#### SECTION 3 - CHEMICAL AND PHYSICAL PROPERTIES

Appearance:	white powder	Boiling Point:
Odor:	none	Melting Point:
pH:	neutral	Specific Gravity:
Water Solubility:	infinite	Vapor Pressure:
Evaporation Rat	e: <u>n/av</u>	Vapor Density:

#### SECTION 4 - FIRE AND EXPLOSION HAZARDS

Flash Point: none

Explosive Limits: none

Extinguishing Media: <u>n/ap</u> <u>Special Firefighting Procedures and Hazards</u>: Material is not combustible. May emit toxic fumes when heated, such as NOx, HCN. HCl. Wear protection as described in Section 6.

## **SECTION 5 - REACTIVITY INFORMATION**

Stable:	<u>X</u>	Unstable:	Precautions: none known	
Hazardous	Polyme	rization: Occ	s: Does Not Occur: <u>X</u>	
ncompatibility:strong acids and oxcidizing agents.				
Hazardous	Decom	position Produc	: When heated, possibly NOx, HCN, HCI.	

FORMULA: n/ap

n/av n/av n/av n/ap n/ap YSI 3682

## SECTION 6 - HEALTH HAZARDS / PROTECTIVE MEASURES / FIRST AID

#### Inhalation:

Possible irritation from dusts. (see CHRONIC below)

Use a NIOSH approved respirator for dusts. Get supplier recommendations. Provide adequate ventilation. Minimize dusty conditions.

Remove to fresh air and provide artificial respiration if needed.

<u>Skin:</u>

Possible irritation from dusts. (see CHRONIC below)

Wear dust-proof gloves and other body protection as needed. Minimize dusty conditions.

Wash exposed areas with soap and water for 15 minutes. Remove contaminated clothing, and wash before re-using.

Eyes:

Possible irritation from dust.

Wear dust barrier goggles. Eliminate dusty conditions.

Flush with water for 15 minutes.

#### **Ingestion**

No effects expected from normal use and minor amounts ingested. Large amounts, over 1 tablespoon, can cause digestive system upset s. (see CHRONIC below)

Reduce dusting. Avoid mouth breathing. Use facemask. Provide adequate ventilation.

Avoid swallowing. Spit out. Drink large amounts of water. Induce vomiting if person is conscious. Otherwise, and if effects persist, get medical attention.

CHRONIC EFFECTS: None reported for this material. "Cyanides" in general are often reported as toxic to humans. Therefore, it is recommended that exposure via skin, inhalation, and ingestion be limited.

#### IN ALL CASES: GET MEDICAL ATTENTION IF EFFECTS PERSIST.

Most likely routes of entry: skin, eyes, ingestion.

#### SECTION 7 - PRECAUTIONS FOR SAFE HANDLING AND USE

<u>Spills and Leaks:</u> Take up powder in any container and hold for disposal. Flush residual to sewer or ground. Provide personal protection as described in Section 6.

<u>Storage and Handling:</u> Keep containers closed. Discard any material that may be contaminated. Minimize dusting.

<u>Waste Disposal</u>: Is not listed as RCRA hazardous waste at this date. Cyanides are restricted in water disposed to streams and to sewers. Therefore, landfill disposal is indicated; check with local disposal companies.

<u>Empty Containers:</u> Rinse well. Dispose as appropriate for glass and plastic containers.

### SECTION 8 - REGULATORY INFORMATION

DOT: Not regulated. SARA Title III, S.313, Form R: Nothing reportable.

The information contained herein is based on data available at this time and is believed to be accurate. However, no warranty is expressed or implied regarding the accuracy of these data or the results to be obtained from the use thereof. Since information contained herein may be applied under conditions beyond our control, and with which we may be unfamiliar, no responsibility is assumed for the results of its use. The person receiving this information shall make his own determination of the suitability of the material for his particular use.

A96008A



MASTER NON HAZARDOUS

Revision Number A96002D, 3/30/06

#### 1. IDENTIFICATION OF THE SUBSTANCE/PREPARATION AND THE COMPANY/UNDERTAKING

Product name	3161 Conductivivty Calibrator 1,000 micromho/cm
Synonyms	None
Chemical characterization	Liquid.
Manufacturer, importer, supplier	YSI, Inc.
	1700/1725 Brannum Lane
	Yellow Springs, OH 45387
	USA
EMERGENCY TELEPHONE	CHEMTREC: 1-800-424-9300

## NUMBER

#### 2. COMPOSITION/INFORMATION ON INGREDIENTS

CAS	Chemical Name	% Weight	ACGIH TWA	Acute toxicity	IARC*	NTP*	OSHA*
7447-40-7	Potassium chloride	0-1	None	NA	N/A	N/A	N/A
7732-18-5	Water	99-100	None	NA	N/A	N/A	N/A

\* IARC - Group 1 (Carcinogenic to humans)

\* NTP - Report on Carcinogens - Known Carcinogens

\* OSHA - Regulated Carcinogens

## 3. HAZARDS IDENTIFICATION

## **Emergency Overview:**

· The product contains no substances which at their given concentration, are considered to be hazardous to health

Eye contact	Can cause severe irritation.	
Skin contact	Exposure can cause skin irritation.	
Inhalation:	Inhalation of dust may cause irritation of respiratory tissue.	
Ingestion:	May be harmful if swallowed.	
General advice	No information available.	
Properties affecting health	No information available	
Principle Routes of Exposure	eyes, absorption, ingestion	

## 4. FIRST AID MEASURES

General advice	<ul> <li>If exposure symptoms persist, seek medical attention.</li> </ul>		
Skin contact	<ul> <li>Wash exposed areas with soap and water for 15 minutes.</li> </ul>		
	<ul> <li>If skin irritation persists, seek medical attention.</li> </ul>		
Eye contact	<ul> <li>Rinse immediately with plenty of water, also under the eyelids, for at least 15 minutes</li> </ul>		
	<ul> <li>If eye irritation persists, seek medical attention</li> </ul>		
Inhalation:	Move to fresh air		
	<ul> <li>If exposure symptoms persist, seek medical attention.</li> </ul>		
Ingestion:	• Do not swallow. Rinse mouth with water and afterwards drink plenty of water.		
	<ul> <li>If effects persist, seek medical attention.</li> </ul>		
Notes to physician	Treat symptomatically		
Protection of first-aiders	Use necessary personal protective equipment		
Aggravated Medical Conditions	<ul> <li>Users with skin conditions (eczema, psoriasis, etc.,) respiratory conditions (asthma, bronchitis, emphysema, etc.,) or with chemical sensitivities should take protective precautions.</li> </ul>		

## MATERIAL SAFETY DATA SHEET MASTER NON HAZARDOUS

HTING MEAS		

Flash point	NA
Suitable extinguishing media	Not applicable to this product
Extinguishing media which must not be used for safety reasons	None
Specific hazabrds	None
Special exposure hazards arising from the substance or preparation itself, its combustion products, or released gases	None
Special protective equipment for firefighters	None
	<ul> <li>As in any fire, wear self-contained breathing apparatus pressure-demand, MSHA/NIOSH (approved or equivalent) and full protective gear</li> </ul>
Specific methods	No special protective measures against fire required
NFPA (National Fire Protection Association)	Health=1 (slight); Reactivity=0, fire=0, & Special = 0 (none)
HMIS (Hazardous Material Information System)	Health=1(slight); Reactivity=0, fire=0, & Special = 0 (none)

Personal precautions	Ensure adequate ventilation	
Environmental precautions	No information available	
Methods for cleaning up	Soak up with inert absorbent material	
	After cleaning, flush away traces with water	

## 7. HANDLING AND STORAGE

## Handling

Technical	<ul> <li>As a rule, at least 10 air changes per hour are recommended at the</li></ul>
measures/Precautions	workplace
Safe handling advice	<ul> <li>Avoid contact with eyes. Wash hands immediately after contact to avoid hand-eye transfer.</li> </ul>

Storage

Technical	<ul> <li>Keep in properly labelled containers</li> </ul>		
measures/Precautions	<ul> <li>Keep containers tightly closed; discard any material that may be</li> </ul>		
	contaminated or, which may have changed composition.		
	<ul> <li>The product is not flammable</li> </ul>		
Incompatible products	Avoid strong acids, oxidizing agents.		
8. EX	POSURE CONTROLS / PERSONAL PROTECTION		
Engineering measures	Ensure eyewash station is readily available		
	<ul> <li>Ensure adequate ventilation, especially in confined areas</li> </ul>		
Personal protective equipment			
Hand protection	Wear appropriate protective gloves		
Eye protection	Avoid contact with eyes		
	<ul> <li>Safety glasses with side-shields or full face shield.</li> </ul>		
Respiratory protection	No information available		
Skin and body protection	lightweight protective clothing		
	boots		
	apron		
Hygiene measures	Handle in accordance with good industrial hygiene and safety practice		
	<ul> <li>Keep away from food, drink and animal feeding stuffs</li> </ul>		

## MATERIAL SAFETY DATA SHEET MASTER NON HAZARDOUS

Environmental exposure controls	No information available	
	9. PHYSICAL AND CHEMICAL PROPERTIES	
General Information		
Form	Liquid.	
Appearance	Clear colorless liquid.	
Odour	None.	
Important Health Safety and E	Environmental Information	
рН	6.50 to 7.50	
Boiling point/range	100°C	
Flash point	Not applicable	
Vapour pressure	equivalent to water.	
Vapour density	equivalent to water vapor.	
Water solubility	Infinitely soluable.	
Specific Gravity	1.00.	
	10. STABILITY AND REACTIVITY	
Stability	Stable under normal conditions.	
Materials to avoid	None. Incompatible with strong acids and oxidizing agents.	
Hazardous decomposition products	None.	
Polymerization	Polymerization does not occur.	
	11. TOXICOLOGICAL INFORMATION	

Acute toxicity

## **Component Information**

## Product Information

Local effects			
Skin irritation	May cause skin irritation in susceptible persons.		
Eye irritation	Dust may cause eye irritation.		
Inhalation:	Inhalation of dust may cause irritation of respiratory tissue.		
Ingestion:	No effects expected from normal use and minor amounts ingested. Ingestion of large amounts (over 1 tablespoon) may cause digestive system upset.		
Sensitization	Not a sensitizer.		
Chronic toxicity	No information available.		
Specific effects			
carcinogenic effects	No information available.		
mutagenic effects	No information available.		
Reproductive toxicity	No information available.		
Target Organ Effects	No information available.		

## 12. ECOLOGICAL INFORMATION

## **Ecotoxicity effects**

## **Component Information**

CAS	Chemical Name	% Weight	ACGIH*
7447-40-7	Potassium chloride	0-1	N/A

#### MATERIAL SAFETY DATA SHEET MASTER NON HAZARDOUS

7700 40 5	NR/- 1	00.400	N1/A
7732-18-5	Water	99-100	N/A

\* ACGIH - Occupational Exposure Limits - TWA's

#### **Product Information**

Aquatic toxicity

No information available.

Not regulated.

### **Other information:**

Ozone depletion potential; ODP; (R-11 = 1)	No information available.
Global warming potential (GWP)	No information available.
Additional ecological information	No information available.
Mobility	No information available
Bioaccumulative potential	No information available
Ecotoxicity effects	No information available
Aquatic toxicity	No information available

#### **13. DISPOSAL CONSIDERATIONS**

Waste from residues / unused products	In accordance with local and national regulations.
Contaminated packaging	Empty containers should be rinsed and disposed of as appropriate for glass and plastic containers

## 14. TRANSPORT INFORMATION

DOT UN-No Proper shipping name Packing group Subsidiary Risk Description

## 15. REGULATORY INFORMATION

#### **U.S.** Inventories

Chemical Name % Weight		ACGIH*	
Potassium chloride	0-1	N/A	
Water	99-100	N/A	
	Potassium chloride	Potassium chloride 0-1	Potassium chloride 0-1 N/A

\* ACGIH - Occupational Exposure Limits - TWA's

#### International Inventories

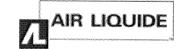
CAS	Chemical Name	% Weight	EUOED*	
7447-40-7	Potassium chloride	0-1	N/A	
7732-18-5	Water	99-100	N/A	
* EUOED - EU Occupational Exposure Directive (98/24/EC) Indicative Occupational Exposure Limit Values (IOELV)				

**16. OTHER INFORMATION** 

Literary reference	None.
Prepared By	YSI, Inc

End of Safety Data Sheet





Material Safety Data Sheets MSDS No: M-704 Date: 04/15/2008

SUPPLIER<br/>ADDRESS:6141 Easton Road, Bldg. 1<br/>PO Box 310EMERGENCY PHONE<br/>NUMBER:(215) 766-8861<br/>NUMBER:Plumsteadville, PA 18949-0310

## **1. CHEMICAL PRODUCT**

PRODUCT ISOBUTYLENE IN AIR NAME:

SYNONYMS: None

2. COMPOSITION, INFORMATION ON INGREDIENTS							
					Exposure Lin	nits (PPM)	
Ingredient Name F	ormula	CAS #	Concentration	ACGIH TLV	OSHA PEL	MAC	Other STEL
ISOBUTYLENE C	C4H8	115-11-7	1-1500 PPM	NE	NE	NE	NE
AIR C	02	132259-10-0	BALANCE	NE	NE	NE	NE

Note: NE = NONE ESTABLISHED

S/A = SIMPLE ASPHYXIANT

## **3. HAZARD INDENTIFICATION**

\* \* \* EMERGENCY OVERVIEW \* \* \* High pressure gas. May accelerate combustion.

#### POTENTIAL HEALTH EFFECTS

ROUTES OF ENTRY: Inhalation

ACUTE EFFECTS: None

CHRONIC EFFECTS: None known

MEDICAL CONDITIONS AGGRAVATED BY OVEREXPOSURE: None known

OTHER EFFECTS OF OVEREXPORSURE: None

CARCINOGENICITY (US ONLY):

NTP - No IARC MONOGRAPHS - No OSHA REGULATED - No

## 4. FIRST AID MEASURES

INHALATION: Immediately remove victim to fresh air. If breathing has stopped, give artificial respiration. If breathing is difficult, give oxygen.

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EYE CONTACT: None

SKIN CONTACT: None

INGESTION: None

IN EVENT OF EXPOSURE, CONSULT A PHYSICIAN

NOTE TO PHYSICIAN: None

## 5. FIRE FIGHTING MEASURES

FLASH POINT: Nonflammable

AUTOIGNITION TEMPERATURE: N/Ap

FLAMMABLE LIMITS: Nonflammable

LOWER: UPPER:

EXTINGUISHING MEDIA: Use what is appropriate for surrounding fire.

SPECIAL FIRE FIGHTING INSTRUCTION AND EQUIPMENT: Wear self-contained breathing apparatus and full protective clothing.Keep fire exposed cylinders cool with water spray.If possible, stop the product flow.

## HAZARDOUS COMBUSTION PRODUCTS: None

UNUSUAL FIRE AND EXPLOSION HAZARDS: Cylinder rupture may occur under fire conditions.Compressed air at high pressure will accelerate the combustion of flammable materials.

## 6. ACCIDENTAL RELEASE MEASURES

CLEAN UP PROCEDURES: Evacuate and ventilate area.Remove leaking cylinder to exhaust hood or safe outdoor area.Shut off source if possible and remove source of heat.

## SPECIALIZED EQUIPMENT: None

## 7. HANDLING AND STORAGE

PRECAUTIONS TO BE TAKEN IN HANDLING: Secure cylinder when using to protect from falling. Use suitable hand truck to move cylinders.

PRECAUTIONS TO BE TAKEN IN STORAGE: Store in well ventilated areas.Keep valve protection cap on cylinders when not in use.

## 8. EXPOSURE CONTROLS / PERSONAL PROTECTION

ENGINEERING CONTROLS: Provide adequate general and local exhaust ventilation.

EYE / FACE PROTECTION: Safety glasses

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SKIN PROTECTION: None

RESPIRATORY PROTECTION: In case of leakage, use self-contained breathing apparatus.

OTHER PROTECTIVE EQUIPMENT: Safety shoes when handling cylinders.

## 9. PHYSICAL AND CHEMICAL PROPERTIES

APPEARANCE: Colorless

ODOR: Odorless

PHYSICAL PRESSURE: Gas

VAPOR PRESSURE: N/Ap

VAPOR DENSITY (AIR=1): 0.991

BOILING POINT (C): N/Ap

SOLUBILITY IN WATER: @20deg.celsius: 18.68cm3/l

SPECIFIC GRAVITY (H2O=1): Gas

**EVAPORATION RATE: Gas** 

ODOR THRESHOLD: N/Ap

## **10. STABILITY AND REACTIVITY**

STABILITY: Stable under normal storage conditions.

CONDITIONS TO AVOID: Storage in poorly ventilated areas. Storage near a heat source.

MATERIALS TO AVOID: Oxidizing agents.

HAZARDOUS POLYMERIZATION: Will not occur.

HAZARDOUS DECOMPOSITION: None

## **11. TOXICOLOGICAL INFORMATION**

LETHAL CONCENTRATION (LC50): NONE ESTABLISHED

LETHAL DOSE 50 (LD50): N/Ap

TERATOGENICITY: N/Ap

REPRODUCTIVE EFFECTS: N/Ap

MUTAGENICITY: N/Ap

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## 12. ECOLOGICAL INFORMATION

No adverse ecological effects are expected.

## 13. DISPOSAL CONSIDERATIONS

WASTE DISPOSAL METHOD: Dispose of non-refillable cylinders in accordance with federal, state and local regulations. Allow gas to vent slowly to atmosphere in an unconfined area or exhaust hood. If the cylinders are the refillable type, return cylinders to supplier with any valve outlet plugs or caps secured and valve protection caps in place.

## 14. TRANSPORT INFORMATION

CONCENTRATION: 1-1500 ppm

DOT DESCRIPTION (US ONLY):

PROPER SHIPPING NAME: Compressed gases, n.o.s. HAZARD CLASS: 2.2 (nonflammable) INDENTIFICATION NUMBER: UN1956 REPORTABLE QUANTITIES: None LABELING: NONFLAMMABLE GAS

ADR / RID (EU Only): Class 2, 1A

SPECIAL PRECAUTIONS: Cylinders should be transported in a secure upright position in a well ventilated truck.

#### **15. REGULATORY INFORMATION**

OSHA: Process Safety Management: Minor component is not listed in appendix A of 29 CFR 1910.119 as a highly hazardous chemical.

TSCA: Mixture is not listed in TSCA inventory.

SARA: The threshold planning quantity for this mixture is 10,000 lbs.

EU NUMBER: N/Ap

NUMBER IN ANNEX 1 OF DIR 67/548: Mixture is not listed in annex 1.

EU CLASSIFICATION: N/Ap

R: 20

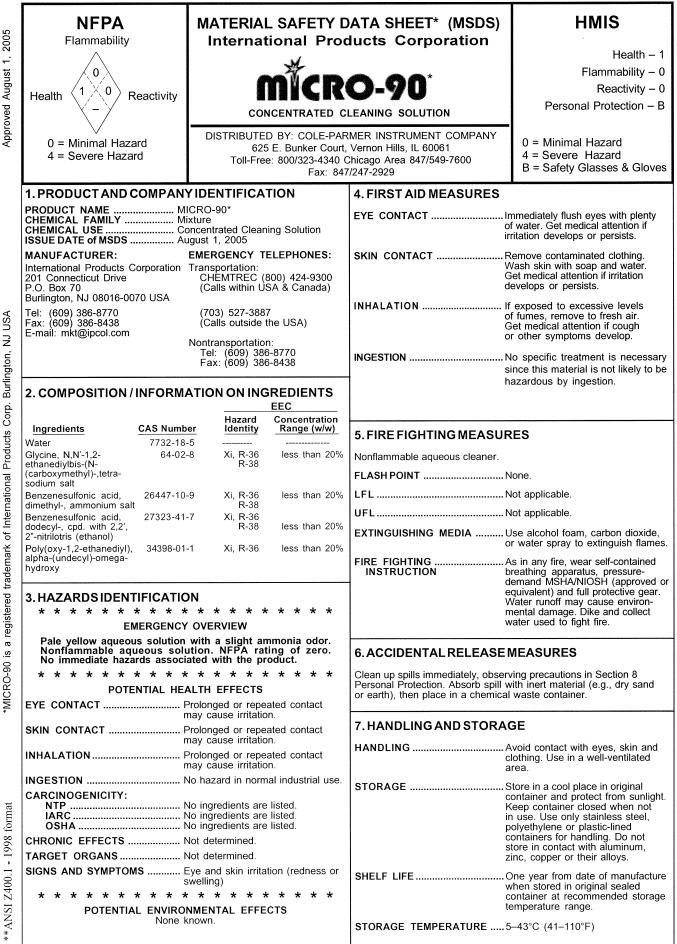
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#### **16. OTHER INFORMATION**

OTHER PRECAUTIONS: Protect containers from physical damage. Do not deface cylinders or labels. Cylinders should be refilled by qualified producers of compressed gas. Shipment of a compressed gas cylinder which has not been filled by the owner or with his written consent is a violation of federal law (49 CFR).

ABBREVIATIONS: N/Ap - Not Applicable N/Av - Not Available SA - Simple Asphyxiant NE - None Established

DISCLAIMER: Information included in this document is given to the best of our knowledge, however, no warranty is made that the information is accurate or complete. We do not accept any responsibility for damages by the use of the document.



8. EXPOSURE CONTROLS / P	EDSONAL DEOTECTION	
		14. TRANSPORTATION INFORMATION
ENGINEERING CONTROLS	should be sufficient to	A.USA
RESPIRATORY PROTECTION	control airborne levels.	D.O.T. SHIPPING Not regulated.
REST INATORY PROTECTION	respiratory protection should be needed.	TECHNICAL SHIPPING NAME . Liquid Detergent
SKIN PROTECTION		D.O.T. LABEL None
	gloves.	D.O.T. PLACARD (non-bulk) None
EYE PROTECTION	Wear safety glasses with sideshields (or goggles). Contact lenses should not	FREIGHT CLASS PACKAGE Class 55 - Liquid Detergent
	be worn.	PRODUCT LABEL Concentrated Cleaning Solution
GENERAL HYGIENE CONSIDERATIONS	There are no known health hazards associated with this	B.CANADA: TDG Not regulated.
	material when used as recommended. The following general hygiene consider- ations are recognized as	C.ENGLAND: APPROVED CARRIAGE LIST Not regulated.
	common, good industrial hygiene practices:	
• Wash hand	Is after use and before eating.	15. REGULATORY INFORMATION
<ul><li>Avoid brea</li><li>Wear safet</li></ul>	thing vapors. ay glasses and gloves.	A. USA
* * * * * * * * * *		TSCA STATUS All ingredients are listed on the
EXPOSURE LIMITS	Not established for product as whole.	TSCA inventory.
Ingredients CAS #	ACGIH	SARA TITLE III, 302/303 EHS None.
None established for individual con	nponents.	SARA TITLE III, 304, HS None.
9. PHYSICAL AND CHEMICAL	PROPERTIES	SARA TITLE III, 313 None.
APPEARANCE	Yellow Liquid.	B. CANADA
ODOR	Ammonia Odor.	DSL All ingredients are listed on the Domestic Substance List.
pH (neat)	ca. 9.5	WHMIS Classification Not controlled.
BOILING POINT	( )	C.EC
FREEZING POINT	ca8°C (18°F)	EINECS All ingredients are listed.
SOLUBILITY IN WATER	•	D.CHIPS Not a significant eye irritant. Not a skin irritant.
SPECIFIC GRAVITY (water = 1) 10. STABILITY AND REACTIVI		
		·
		16. STATE REGULATORY INFORMATION
HAZARDOUS POLYMERIZATION		For details on specific requirements, you should contact the
INCOMPATIBILITIES	. May etch aluminum and zinc. Do not mix with other cleaners. Mixing with chlorine-based cleaners may produce toxic gasses.	appropriate agency in your state.
DECOMPOSITION PRODUCTS	-	17. OTHER INFORMATION
11. TOXICOLOGICAL INFORM		PREPARED BY K. Wyrofsky, Vice President of Marketing
Eye: Irritant per USA-FHSA criteria.	no No. 405	APPROVED BY T. McGuckin, Vice President of Quality & Safety
Not an Irritant per OECD Guideli Skin: Not an irritant per USA-FHSA cr	iteria.	APPROVAL DATE August 1, 2005
Not an irritant per OECD Guideli Dral: LD <sub>50</sub> is greater than 5g/kg (rats).		NOTE: All data presented here are for the full-strength product unless otherwise noted. However, recommended usage is as a
		1-2% w/w solution in water.
Contains no CFCs, ODCs, phosphates, silicates, borates, halogens, or phenols. <b>13. DISPOSAL CONSIDERATIONS</b> MICRO-90* is not considered a hazardous waste under Federal Hazardous Waste Regulations 40 CFR 261. Please be advised,		While International Products Corporation believes th information contained herein to be true and accurate, it ha relied on information provided by others. International Product
		Corporation makes no warranties, express or implied, as t the accuracy or adequacy of the information contained herei or with respect to the results to be obtained from the use of
		or with respect to the results to be obtained from the use of the product. International Products Corporation disclaims a liability with respect to the use of this product, including withou limitation, liability for injury to the user or third-party persons.
Note: Chemical additions, processing or nay make the waste managem n this MSDS incomplete, inaccurate or	ent information presented	
	,	05070

## Alconox ${\mathbb R}$

## MATERIAL SAFETY DATA SHEET

Alconox, Inc.

9 East 40th Street, Suite 200 New York, NY 10016

24 Hour Emergency Number - Chem-Tel (800) 255-3924

## I. IDENTIFICATION

Product Name (as appears on label)	ALCONOX (C-P# 17775-00)	
CAS Registry Number:	Not Applicable	
Effective Date:	July 9, 1999	
Chemical Family:	Anionic Powdered Detergent	

#### **II. HAZARDOUS INGREDIENTS/IDENTITY INFORMATION**

There are no hazardous ingredients in ALCONOX as defined by the OSHA Standard and Hazardous Substance List 29 CFR 1910 Subpart Z.

Boiling Point (F):	Not Applicable		
Vapor Pressure (mm Hg):	Not Applicable		
Vapor Density (AIR=1):	Not Applicable		
Specific Gravity (Water=1):	Not Applicable		
Melting Point:	Not Applicable		
Evaporation Rate (Butyl Acetate=1):	Not Applicable		
Solubility in Water:	Appreciable-Soluble to 10% at ambient conditions		
Appearance:	White powder interspersed with cream colored flakes.		

## III. PHYSICAL/CHEMICAL CHARACTERISTICS

#### IV. FIRE AND EXPLOSION DATA

Flash Point (Method Used):	None
Flammable Limits:	LEL: No Data UEL: No Data
Extinguishing Media:	Water, dry chemical, CO <sub>2</sub> , foam
Eiretionting	Self-contained positive pressure breathing apparatus and protective clothing should be worn when fighting fires involving chemicals.
Unusual Fire and Explosion Hazards:	None

## V. REACTIVITY DATA

Stability:	Stable
Hazardous Polymerization:	Will not occur

Incompatibility (Materials to Avoid):	None
Hazardous Decomposition or Byproducts:	May release CO <sub>2</sub> on burning

## VI. HEALTH HAZARD DATA

Route(s) of Entry:	Inhalation? Yes Skin? No Ingestion? Yes
Health Hazards (Acute and Chronic):	Inhalation of powder may prove locally irritating to mucous membranes. Ingestion may cause discomfort and/or diarrhea. Eye contact may prove irritating.
	NTP? No IARC Monographs? No OSHA Regulated? No
Signs and Symptoms of Exposure:	Exposure may irritate mucous membranes. May cause sneezing.
5 00	Not established. Unnecessary exposure to this product or any industrial chemical should be avoided. Respiratory conditions may be aggravated by powder.
Emergency and First Aid Procedures:	Eyes: Immediately flush eyes with water for at least 15 minutes. Call a physician. Skin: Flush with plenty of water. Ingestion: Drink large quantities of water or milk. Do not induce vomiting. If vomiting occurs readminister fluids. See a physician for discomfort.

## VII. PRECAUTIONS FOR SAFE HANDLING AND USE

Motorial ic Released or	Material foams profusely. Recover as much as possible and flush remainder to sewer. Material is biodegradable.
Waste Disposal Method:	Small quantities may be disposed of in sewer. Large quantities should be disposed of in accordance with local ordinances for detergent products.
Precautions to be Taken in Storing and Handling:	Material should be stored in a dry area to prevent caking.
	No special requirements other than the good industrial hygiene and safety practices employed with any industrial chemical.

## VIII. CONTROL MEASURES

Respiratory Protection (Specify Type):	Dust mask - Recommended
Ventilation:	Local Exhaust-Normal Special-Not Required Mechanical-Not Required Other-Not Required
Protective Gloves:	Impervious gloves are useful but not required.
Eye Protection:	Goggles are recommended when handling solutions.
Other Protective Clothing or Equipment:	None
Work/Hygienic Practices:	Wash hands before eating, drinking or smoking.

THE INFORMATION HEREIN IS GIVEN IN GOOD FAITH BUT NO WARRANTY IS EXPRESSED OR IMPLIED.

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Material Safety Data Sheet Collection	Unleaded Petrol
Genium group inc. 1171 RiverFront Center, Amsterdam, NY 12010 (518) 842-4111	AUT5000 Issue Date: 2006-06
Section 1 - Chemical Pro	oduct and Company Identification 61
Material Name: Unleaded Petrol Chemical Formula: Mixture of hydrocarbons EINECS Number: 232-349-1 ACX Number: X1003056-5 Synonyms: AUTOMOTIVE GASOLINE, LEAD-FR NATURAL GASOLINE; PETROL; UNLEADED P General Use: Lead free motor fuel for internal combu	
Section 2 - Compositi	on / Information on Ingredients
Name gasoline benzene	CAS         %           8006-61-9         >90           71-43-2         5 max.
OSHA PEL NIOSH RE	EL
ACGIH TLV TWA: 300 ppm, 890 mg/m <sup>3</sup> ; STEL: 500 ppm, 1480 mg/m <sup>3</sup> . Section 3 - I Flammability Toxicity Body Contact Reactivity Chronic 0 1 Min Low ANSI Signal Word Danger!	Hazards Identification         Identification
	<b>rgency Overview</b> ☆☆☆☆☆ n/respiratory tract. Other Acute Effects: dizziness, drunkenness,
Potent Target Organs: skin, eye, respiratory system, central Primary Entry Routes: inhalation, ingestion, skin co	

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#### Acute Effects

Inhalation: The vapor is discomforting to the upper respiratory tract and may be harmful if exposure is prolonged. Inhalation hazard is increased at higher temperatures. Acute effects from inhalation of high concentrations of vapor are pulmonary irritation, including coughing, with nausea; central nervous system depression - characterized by headache and dizziness, increased reaction time, fatigue and loss of coordination. If exposure to highly concentrated solvent atmosphere is prolonged this may lead to narcosis, unconsciousness, even coma and possible death. WARNING: Intentional misuse by concentrating/inhaling contents may be lethal. High inhaled concentrations of mixed hydrocarbons may produce narcosis characterized by nausea, vomiting and lightheadedness. Inhalation of aerosols may produce severe pulmonary edema, pneumonitis and pulmonary hemorrhage. Inhalation of petroleum hydrocarbons consisting substantially of low molecular weight species may produce irritation of mucous membranes, incoordination, giddiness, nausea, vertigo, confusion, headache, appetite loss, drowziness, tremors and anesthetic stupor. Massive exposures may produce central nervous system depression with sudden collapse and deep coma: fatalities have been recorded. Irritation of the brain and/or apneic anoxia may produce convulsions. Although recovery following overexposure is generally complete, cerebral micro- hemorrhage of focal post-inflammatory scarring may produce eleptiform seizures some months after the exposure. Pulmonary episodes may include chemical pneumonitis with edema and hemorrhage. The lighter hydrocarbons may produce kidney and neurotoxic effects. Liquid paraffins may produce anesthesia and depressant actions leading to weakness, dizziness, slow and shallow respiration, unconsciousness, convulsions and death. C<sub>5.7</sub> paraffins may also produce polyneuropathy. Aromatic hydrocarbons accumulate in lipid-rich tissues (typically the brain, spinal cord and peripheral nerves) and may produce functional impairment manifested by nonspecific symptoms such as nausea, weakness, fatigue, vertigo; severe exposures may produce inebriation or unconsciousness. Many of the petroleum hydrocarbons are cardiac sensitizers and may cause ventricular fibrillations.

- **Eye:** The liquid may produce eye discomfort and is capable of causing temporary impairment of vision and/or transient eye inflammation, ulceration. The vapor is discomforting to the eyes. Petroleum hydrocarbons may produce pain after direct contact with the eyes. Slight, but transient, disturbances of the corneal epithelium may also result. The aromatic fraction may produce irritation and lachrymation. The material may produce moderate eye irritation leading to inflammation. Repeated or prolonged exposure to irritants may produce conjunctivitis.
- **Skin:** The material is moderately discomforting to the skin if exposure is prolonged. The material contains a component that may be absorbed through the skin and may cause drying of the skin, which may lead to dermatitis from repeated exposures over long periods. Toxic effects may result from skin absorption. Open cuts, abraded or irritated skin should not be exposed to this material. The material may accentuate any pre-existing dermatitis condition.
- **Ingestion:** Considered an unlikely route of entry in commercial/industrial environments. The liquid may produce gastrointestinal discomfort and may be harmful if swallowed. Ingestion may result in nausea, pain and vomiting. Vomit entering the lungs by aspiration may cause potentially lethal chemical pneumonitis. Ingestion of petroleum hydrocarbons may produce irritation of the pharynx, esophagus, stomach and small intestine with edema and mucosal ulceration. Resulting symptoms include a burning sensation in the mouth and throat. Large amounts may produce narcosis with nausea and vomiting, weakness or dizziness, slow and shallow respiration, swelling of the abdomen, unconsciousness and convulsions. Myocardial injury may produce arrhythmias, ventricular fibrillation and electrocardiographic changes. Central nervous system depression may also occur. Light aromatic hydrocarbons produce a warm, sharp, tingling sensation on contact with taste buds and may anesthetize the tongue. Aspiration into the lungs may produce coughing, gagging, and a chemical pneumonitis with pulmonary edema and hemorrhage.

**Carcinogenicity:** NTP - Not listed; IARC - Group 2B, Possibly carcinogenic to humans; OSHA - Not listed; NIOSH - Listed as carcinogen; ACGIH - Class A3, Animal carcinogen; EPA - Not listed; MAK - Not listed.

**Chronic Effects:** Chronic solvent inhalation exposures may result in nervous system impairment and liver and blood changes. Prolonged or continuous skin contact with the liquid may cause defatting with drying, cracking, irritation and dermatitis following. Chronic poisoning may occur from vapor inhalation or skin absorption. The most significant toxic effect is insidious and irreversible injury to the blood-forming tissue by benzene. Leukemia may develop. Chronic exposure may cause headache, fatigue, loss of appetite and lassitude with incipient blood effects including anemia and blood changes. Gasoline "sniffing" has caused severe nerve damage. Repeated or prolonged exposure to mixed hydrocarbons may produce narcosis with dizziness, weakness, irritability, concentration and/or memory loss, tremor in the fingers and tongue, vertigo, olfactory disorders, constriction of visual field, paresthesias of the extremities, weight loss and anemia and degenerative changes in the liver and kidney. Chronic exposure by petroleum workers to the lighter hydrocarbons has been associated with visual disturbances, damage to the central nervous system, peripheral neuropathies (including numbness and paresthesias), psychological and neurophysiological deficits, bone marrow toxicities (including hypoplasia, possibly due to benzene) and hepatic and renal involvement. Chronic dermal exposure to petroleum hydrocarbons may result in defatting which produces localized dermatoses. Surface cracking and erosion may also increase susceptibility to infection by microorganisms.

2006-06

**Unleaded Petrol** 

**AUT5000** 

#### **Section 4 - First Aid Measures** Inhalation: Remove to fresh air. Lay patient down. Keep warm and rested. ວັອອ If breathing is shallow or has stopped, ensure clear airway and apply resuscitation. Transport to DOL hospital, or doctor. Eye Contact: Immediately hold the eyes open and wash continuously for at least 15 minutes with erc fresh running water. Ensure irrigation under eyelids by occasionally lifting the upper and lower lids. Transport to hospital or doctor without delay. Removal of contact lenses after an eye injury should only be undertaken by skilled personnel. Skin Contact: Immediately remove all contaminated clothing, including footwear (after rinsing with water). Wash affected areas thoroughly with water (and soap if available). Seek medical attention in event of irritation. Ingestion: Contact a Poison Control Center. If swallowed, do NOT induce vomiting. Give a glass of water. After first aid, get appropriate in-plant, paramedic, or community medical support. **Note to Physicians:** For acute or short term repeated exposures to petroleum distillates or related hydrocarbons: 1. Primary threat to life from pure petroleum distillate ingestion and/or inhalation is respiratory failure. 2. Patients should be quickly evaluated for signs of respiratory distress (e.g. cyanosis, tachypnea, intercostal retraction, obtundation) and given oxygen. Patients with inadequate tidal volumes or poor arterial blood gases (pO, <50 mm Hg or pCO, >50 mm Hg) should be intubated. 3. Arrhythmias complicate some hydrocarbon ingestion and/or inhalation and electrocardiographic evidence of myocardial injury has been reported; intravenous lines and cardiac monitors should be established in obviously symptomatic patients. The lungs excrete inhaled solvents, so that hyperventilation improves clearance. 4. A chest x-ray should be taken immediately after stabilization of breathing and circulation to document aspiration and detect the presence of pneumothorax. 5. Epinephrine (adrenalin) is not recommended for treatment of bronchospasm because of potential myocardial sensitization to catecholamines. Inhaled cardioselective bronchodilators (e.g. Alupent, Salbutamol) are the preferred agents, with aminophylline a second choice. 6. Lavage is indicated in patients who require decontamination; ensure use of cuffed endotracheal tube in adult patients. **Section 5 - Fire-Fighting Measures** Flash Point: -43 °C See Autoignition Temperature: 280 °C DOL **LEL:** 1.4% v/v 6 **UEL:** 7.6% v/v ERC Extinguishing Media: Foam. Dry chemical powder. 1 0 Bromochlorodifluoromethane (BCF) (where regulations permit). Carbon dioxide. General Fire Hazards/Hazardous Combustion Products: Liquid and vapor are highly flammable. Severe fire hazard when exposed to heat, flame and/or oxidizers. Vapor forms an explosive mixture with air. Severe explosion hazard, in the form of vapor, when exposed to flame or spark. Vapor may travel a considerable distance to source of ignition. Heating may cause expansion/decomposition with violent rupture of containers. On Fire Diamond combustion, may emit toxic fumes of carbon monoxide (CO). Fire Incompatibility: Avoid contamination with oxidizing agents, i.e. nitrates, oxidizing acids, chlorine bleaches, pool chlorine etc., as ignition may result. Fire-Fighting Instructions: Alert fire department and tell them location and nature of hazard. May be violently or explosively reactive. Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or water ways. If safe, switch off electrical equipment until vapour fire hazard removed. Use water delivered as a fine spray to control fire and cool adjacent area. Avoid spraying water onto liquid pools. Do not approach containers suspected to be hot. Cool fire exposed containers with water spray from a protected location. If safe to do so, remove containers from path of fire. **Section 6 - Accidental Release Measures** Small Spills: Remove all ignition sources. Clean up all spills immediately. Avoid breathing vapors วัยย and contact with skin and eyes. Control personal contact by using protective equipment. Contain and absorb small quantities with vermiculite or other absorbent material. Wipe up. Collect residues in a DOL flammable waste container. ERC Large Spills: Clear area of personnel and move upwind. Alert fire department and tell them location and nature of hazard. May be violently or explosively reactive. Wear breathing apparatus plus protective gloves. Prevent, by any means available, spillage from entering drains or water ways. No smoking, naked lights or ignition sources. Increase ventilation. Stop leak if safe to do so. Water spray or fog may be used to disperse/absorb vapor. Contain spill with sand, earth or vermiculite. Use only

## **Unleaded Petrol**

AUT5000

spark-free shovels and explosion proof equipment. Collect recoverable product into labeled containers for recycling. Absorb remaining product with sand, earth or vermiculite. Collect solid residues and seal in labelled drums for disposal. Wash area and prevent runoff into drains.

If contamination of drains or waterways occurs, advise emergency services.

Regulatory Requirements: Follow applicable OSHA regulations (29 CFR 1910.120).

## Section 7 - Handling and Storage

**Handling Precautions:** Avoid generating and breathing mist. Avoid all personal contact, including inhalation. Wear protective clothing when risk of exposure occurs. Use in a well-ventilated area. Prevent concentration in hollows and sumps. DO NOT enter confined spaces until atmosphere has been checked. Avoid smoking, bare lights, heat or ignition sources. When handling, DO NOT eat, drink or smoke. Vapor may ignite on pumping or pouring due to static electricity. DO NOT use plastic buckets. Ground and secure metal containers when dispensing or pouring product. Use spark-free tools when handling. Avoid contact with incompatible materials. Keep containers securely sealed. Avoid physical damage to containers. Always wash hands with soap and water after handling. Work clothes should be laundered separately. Use good occupational work practices. Observe manufacturer's storing and handling recommendations. Atmosphere should be regularly checked against established exposure standards to ensure safe working conditions.

**Recommended Storage Methods:** Metal can, metal drum. Packing as recommended by manufacturer. Check all containers are clearly labeled and free from leaks.

Regulatory Requirements: Follow applicable OSHA regulations.

## Section 8 - Exposure Controls / Personal Protection

**Engineering Controls:** CARE: Use of a quantity of this material in confined space or poorly ventilated area, where rapid build-up of concentrated atmosphere may occur, could require increased ventilation and/or protective gear. Use in a well-ventilated area. If inhalation risk of overexposure exists, wear a NIOSH approved organic-vapor respirator. Correct respirator fit is essential to obtain adequate protection. In confined spaces where there is inadequate ventilation, wear full-face air supplied breathing apparatus. Provide adequate ventilation in warehouse or closed storage areas.

#### Personal Protective Clothing/Equipment:

Eyes: Safety glasses with side shields; or as required, chemical goggles.

Contact lenses pose a special hazard; soft lenses may absorb irritants and all lenses concentrate them.

Hands/Feet: Barrier cream with polyethylene gloves or PVC gloves. Safety footwear. Do NOT use this product to clean the skin.

#### **Respiratory Protection:**

Exposure Range >300 to 1000 ppm: Air Purifying, Negative Pressure, Half Mask

Exposure Range >1000 to 15,000 ppm: Air Purifying, Negative Pressure, Full Face

Exposure Range >15,000 to 300,000 ppm: Supplied Air, Constant Flow/Pressure Demand, Full Face

Exposure Range >300,000 to unlimited ppm: Self-contained Breathing Apparatus, Pressure Demand, Full Face Cartridge Color: black

**Other:** Overalls. Ensure that there is ready access to eye wash unit. Ensure there is ready access to an emergency shower.

## Section 9 - Physical and Chemical Properties

**Appearance/General Info:** Purple, highly flammable, volatile liquid with characteristic sharp odor. Floats on water. Consists of a complex mixture of hydrocarbons with small amounts of residual benzene from the refining operations.

Physical State: Liquid Odor Threshold: 0.005 ppm Vapor Pressure (kPa): 53.33 at 20 °C Vapor Density (Air=1): > 2 Formula Weight: Not applicable. Specific Cravity (HeO=1, at 4 °C): 0.72 0.735

**Specific Gravity (H<sub>2</sub>O=1, at 4 °C):** 0.72-0.735 at 15 °C **Evaporation Rate:** Fast

pH: Not applicable
pH (1% Solution): Not applicable.
Boiling Point: 38.89 °C (102 °F)
Freezing/Melting Point: Not available
Volatile Component (% Vol): 100
Decomposition Temperature (°C): Not available.
Water Solubility: Insoluble

## Section 10 - Stability and Reactivity

**Stability/Polymerization/Conditions to Avoid:** Presence of incompatible materials. Product is considered stable. Hazardous polymerization will not occur.

Storage Incompatibilities: Avoid storage with oxidizers.

2006-06

## **Unleaded Petrol**

**AUT5000** 

Section 11 - Toxicological Information

**Toxicity** 

Oral (rat) LD<sub>50</sub>: 18800 mg/kg

Irritation

Skin (rabbit): 500 mg/24h mild

## Section 12 - Ecological Information

Environmental Fate: No data found. Ecotoxicity: No data found.

**Biochemical Oxygen Demand (BOD):** 8%, 5 days

## Section 13 - Disposal Considerations

**Disposal:** Consult manufacturer for recycling options and recycle where possible. Follow all applicable federal, state, and local laws. Incinerate residue at an approved site. Recycle containers where possible, or dispose of in an authorized landfil.

BEWARE: Empty solvent, paint, lacquer and flammable liquid drums present a severe explosion hazard if cut by flame torch or welded. Even when thoroughly cleaned or reconditioned, the drum seams may retain sufficient solvent to generate an explosive atmosphere in the drum.

## Section 14 - Transport Information

## DOT Hazardous Materials Table Data (49 CFR 172.101):

Shipping Name and Description: GasolineID: UN1203Hazard Class: 3 - Flammable and combustible liquidPacking Group: II - Medium DangerSymbols:Label Codes: 3 - Flammable LiquidSpecial Provisions: 139, B33, B101, T8Packaging:Exceptions: 150 Non-bulk: 202 Bulk: 242Quantity Limitations:Passenger aircraft/rail: 5 LCargo aircraft only: 60 LVessel Stowage:Location: EOther:

## **Section 15 - Regulatory Information**

EPA Regulations: RCRA 40 CFR: Not listed CERCLA 40 CFR 302.4: Not listed SARA 40 CFR 372.65: Not listed SARA EHS 40 CFR 355: Not listed TSCA: Listed

## Section 16 - Other Information

**Disclaimer:** Judgments as to the suitability of information herein for the purchaser's purposes are necessarily the purchaser's responsibility. Although reasonable care has been taken in the preparation of such information, Genium Group, Inc. extends no warranties, makes no representations, and assumes no responsibility as to the accuracy or suitability of such information for application to the purchaser's intended purpose or for consequences of its use.





SECTION 1 PRODUCT AND COMPANY IDENTIFICATION

# Havoline® Motor Oil (Deposit Shield)

**Product Use:** Engine Oil **Product Number(s):** CPS223391, CPS223392, CPS223393, CPS223394, CPS223395, CPS223396, CPS223397

**Synonyms:** Havoline® Motor Oil SAE 10W-30, Havoline® Motor Oil SAE 10W-40, Havoline® Motor Oil SAE 20W-50, Havoline® Motor Oil SAE 30, Havoline® Motor Oil SAE 40, Havoline® Motor Oil SAE 5W-20, Havoline® Motor Oil SAE 5W-30

#### **Company Identification**

Chevron Products Company Global Lubricants 6001 Bollinger Canyon Road San Ramon, CA 94583 United States of America

#### **Transportation Emergency Response**

CHEMTREC: (800) 424-9300 or (703) 527-3887

#### Health Emergency

Chevron Emergency Information Center: Located in the USA. International collect calls accepted. (800) 231-0623 or (510) 231-0623

# Product Information

email : lubemsds@chevrontexaco.com Product Information: 800-LUBE-TEK MSDS Requests: 800-414-6737

## SECTION 2 COMPOSITION/ INFORMATION ON INGREDIENTS

COMPONENTS	CAS NUMBER	AMOUNT
Highly refined mineral oil (C15 - C50)	Mixture	70 - 95 %weight

SECTION 3 HAZARDS IDE	TIFICATION	

#### IMMEDIATE HEALTH EFFECTS

Eye: Not expected to cause prolonged or significant eye irritation.

**Skin:** Contact with the skin is not expected to cause prolonged or significant irritation. Contact with the skin is not expected to cause an allergic skin response. Not expected to be harmful to internal organs if absorbed through the skin.

Ingestion: Not expected to be harmful if swallowed.

**Inhalation:** Not expected to be harmful if inhaled. Contains a petroleum-based mineral oil. May cause respiratory irritation or other pulmonary effects following prolonged or repeated inhalation of oil mist at airborne levels above the recommended mineral oil mist exposure limit. Symptoms of respiratory irritation may include coughing and difficulty breathing.

#### SECTION 4 FIRST AID MEASURES

**Eye:** No specific first aid measures are required. As a precaution, remove contact lenses, if worn, and flush eyes with water.

**Skin:** No specific first aid measures are required. As a precaution, remove clothing and shoes if contaminated. To remove the material from skin, use soap and water. Discard contaminated clothing and shoes or thoroughly clean before reuse.

**Ingestion:** No specific first aid measures are required. Do not induce vomiting. As a precaution, get medical advice.

**Inhalation:** No specific first aid measures are required. If exposed to excessive levels of material in the air, move the exposed person to fresh air. Get medical attention if coughing or respiratory discomfort occurs.

SECTION 5 FIRE FIGHTING ME	

#### FIRE CLASSIFICATION:

OSHA Classification (29 CFR 1910.1200): Not classified by OSHA as flammable or combustible.

NFPA RATINGS: Health: 0 Flammability: 1 Reactivity: 0

#### FLAMMABLE PROPERTIES:

Flashpoint: (Cleveland Open Cup) 200 °C (392 °F) (Min)

Autoignition: No Data Available

Flammability (Explosive) Limits (% by volume in air): Lower: Not Applicable Upper: Not Applicable

**EXTINGUISHING MEDIA:** Use water fog, foam, dry chemical or carbon dioxide (CO2) to extinguish flames.

#### **PROTECTION OF FIRE FIGHTERS:**

**Fire Fighting Instructions:** This material will burn although it is not easily ignited. For fires involving this material, do not enter any enclosed or confined fire space without proper protective equipment, including self-contained breathing apparatus.

**Combustion Products:** Highly dependent on combustion conditions. A complex mixture of airborne solids, liquids, and gases including carbon monoxide, carbon dioxide, and unidentified organic compounds will be evolved when this material undergoes combustion.

## SECTION 6 ACCIDENTAL RELEASE MEASURES

Protective Measures: Eliminate all sources of ignition in vicinity of spilled material.

**Spill Management:** Stop the source of the release if you can do it without risk. Contain release to prevent further contamination of soil, surface water or groundwater. Clean up spill as soon as possible, observing precautions in Exposure Controls/Personal Protection. Use appropriate techniques such as applying non-combustible absorbent materials or pumping. Where feasible and appropriate, remove contaminated soil. Place contaminated materials in disposable containers and dispose of in a manner consistent with applicable regulations.

**Reporting:** Report spills to local authorities and/or the U.S. Coast Guard's National Response Center at (800) 424-8802 as appropriate or required.

#### SECTION 7 HANDLING AND STORAGE

#### Precautionary Measures: Keep out of the reach of children.

**General Handling Information:** Avoid contaminating soil or releasing this material into sewage and drainage systems and bodies of water.

**Static Hazard:** Electrostatic charge may accumulate and create a hazardous condition when handling this material. To minimize this hazard, bonding and grounding may be necessary but may not, by themselves, be sufficient. Review all operations which have the potential of generating and accumulating an electrostatic charge and/or a flammable atmosphere (including tank and container filling, splash filling, tank cleaning, sampling, gauging, switch loading, filtering, mixing, agitation, and vacuum truck operations) and use appropriate mitigating procedures. For more information, refer to OSHA Standard 29 CFR 1910.106, 'Flammable and Combustible Liquids', National Fire Protection Association (NFPA 77, 'Recommended Practice on Static Electricity', and/or the American Petroleum Institute (API) Recommended Practice 2003, 'Protection Against Ignitions Arising Out of Static, Lightning, and Stray Currents'.

**Container Warnings:** Container is not designed to contain pressure. Do not use pressure to empty container or it may rupture with explosive force. Empty containers retain product residue (solid, liquid, and/or vapor) and can be dangerous. Do not pressurize, cut, weld, braze, solder, drill, grind, or expose such containers to heat, flame, sparks, static electricity, or other sources of ignition. They may explode and cause injury or death. Empty containers should be completely drained, properly closed, and promptly returned to a drum reconditioner or disposed of properly.

## SECTION 8 EXPOSURE CONTROLS/PERSONAL PROTECTION

#### **GENERAL CONSIDERATIONS:**

Consider the potential hazards of this material (see Section 3), applicable exposure limits, job activities, and other substances in the work place when designing engineering controls and selecting personal protective equipment. If engineering controls or work practices are not adequate to prevent exposure to harmful levels of this material, the personal protective equipment listed below is recommended. The user should read and understand all instructions and limitations supplied with the equipment since protection is usually provided for a limited time or under certain circumstances.

## ENGINEERING CONTROLS:

Use in a well-ventilated area.

#### PERSONAL PROTECTIVE EQUIPMENT

**Eye/Face Protection:** No special eye protection is normally required. Where splashing is possible, wear safety glasses with side shields as a good safety practice.

**Skin Protection:** No special protective clothing is normally required. Where splashing is possible, select protective clothing depending on operations conducted, physical requirements and other substances in the workplace. Suggested materials for protective gloves include: 4H (PE/EVAL), Nitrile Rubber, Silver Shield, Viton.

Respiratory Protection: No respiratory protection is normally required.

If user operations generate an oil mist, determine if airborne concentrations are below the occupational exposure limit for mineral oil mist. If not, wear an approved respirator that provides adequate protection from the measured concentrations of this material. For air-purifying respirators use a particulate cartridge.

Use a positive pressure air-supplying respirator in circumstances where air-purifying respirators may not provide adequate protection.

#### **Occupational Exposure Limits:**

Component	Agency	TWA	STEL	Ceiling	Notation
Highly refined mineral oil (C15 - C50)	ACGIH	5 mg/m3	10 mg/m3		
Highly refined mineral oil (C15 - C50)	OSHA Z-1	5 mg/m3			

#### SECTION 9 PHYSICAL AND CHEMICAL PROPERTIES

Attention: the data below are typical values and do not constitute a specification.

Color: Amber Physical State: Liquid Odor: Petroleum odor pH: Not Applicable Vapor Pressure: <0.01 mmHg @ 100 °C (212 °F) Vapor Density (Air = 1): >1 Boiling Point: >315°C (599°F) Solubility: Soluble in hydrocarbons; insoluble in water Freezing Point: Not Applicable Specific Gravity: 0.87 @ 15.6°C (60.1°F) / 15.6°C (60.1°F) (Typical) Density: 0.866 kg/l @ 15°C (59°F) (Typical) Viscosity: 7.6 mm2/s @ 100°C (212°F) (Min)

#### SECTION 10 STABILITY AND REACTIVITY

**Chemical Stability:** This material is considered stable under normal ambient and anticipated storage and handling conditions of temperature and pressure.

**Incompatibility With Other Materials:** May react with strong acids or strong oxidizing agents, such as chlorates, nitrates, peroxides, etc.

Hazardous Decomposition Products: None known (None expected)

Hazardous Polymerization: Hazardous polymerization will not occur.

#### SECTION 11 TOXICOLOGICAL INFORMATION

#### IMMEDIATE HEALTH EFFECTS

**Eye Irritation:** The eye irritation hazard is based on evaluation of data for similar materials or product components.

**Skin** Irritation: The skin irritation hazard is based on evaluation of data for similar materials or product components.

Skin Sensitization: The skin sensitization hazard is based on evaluation of data for similar materials or product components.

**Acute Dermal Toxicity:** The acute dermal toxicity hazard is based on evaluation of data for similar materials or product components.

Acute Oral Toxicity: The acute oral toxicity hazard is based on evaluation of data for similar materials or product components.

**Acute Inhalation Toxicity:** The acute inhalation toxicity hazard is based on evaluation of data for similar materials or product components.

## ADDITIONAL TOXICOLOGY INFORMATION:

This product contains petroleum base oils which may be refined by various processes including severe solvent extraction, severe hydrocracking, or severe hydrotreating. None of the oils requires a cancer warning under the OSHA Hazard Communication Standard (29 CFR 1910.1200). These oils have not been listed in the National Toxicology Program (NTP) Annual Report nor have they been classified by the International Agency for Research on Cancer (IARC) as; carcinogenic to humans (Group 1), probably carcinogenic to humans (Group 2A), or possibly carcinogenic to humans (Group 2B). These oils have not been classified by the American Conference of Governmental Industrial Hygienists (ACGIH) as: confirmed human carcinogen (A1), suspected human carcinogen (A2), or confirmed animal carcinogen

with unknown relevance to humans (A3).

During use in engines, contamination of oil with low levels of cancer-causing combustion products occurs. Used motor oils have been shown to cause skin cancer in mice following repeated application and continuous exposure. Brief or intermittent skin contact with used motor oil is not expected to have serious effects in humans if the oil is thoroughly removed by washing with soap and water.

## SECTION 12 ECOLOGICAL INFORMATION

#### ECOTOXICITY

This material is not expected to be harmful to aquatic organisms.

#### ENVIRONMENTAL FATE

This material is not expected to be readily biodegradable.

## SECTION 13 DISPOSAL CONSIDERATIONS

Use material for its intended purpose or recycle if possible. Oil collection services are available for used oil recycling or disposal. Place contaminated materials in containers and dispose of in a manner consistent with applicable regulations. Contact your sales representative or local environmental or health authorities for approved disposal or recycling methods.

#### SECTION 14 TRANSPORT INFORMATION

The description shown may not apply to all shipping situations. Consult 49CFR, or appropriate Dangerous Goods Regulations, for additional description requirements (e.g., technical name) and mode-specific or quantity-specific shipping requirements.

**DOT Shipping Description:** PETROLEUM LUBRICATING OIL, NOT REGULATED AS A HAZARDOUS MATERIAL FOR TRANSPORTATION UNDER 49 CFR **Additional Information:**NOT HAZARDOUS BY U.S. DOT. ADR/RID HAZARD CLASS NOT APPLICABLE.

IMO/IMDG Shipping Description: PETROLEUM LUBRICATING OIL; NOT REGULATED AS DANGEROUS GOODS FOR TRANSPORT UNDER THE IMDG CODE

ICAO/IATA Shipping Description: PETROLEUM LUBRICATING OIL; NOT REGULATED AS DANGEROUS GOODS FOR TRANSPORT UNDER ICAO

## SECTION 15 REGULATORY INFORMATION

#### EPCRA 311/312 CATEGORIES:

1.Immediate (Acute) Health Effects:NO2.Delayed (Chronic) Health Effects:NO3.Fire Hazard:NO4.Sudden Release of Pressure Hazard:NO5.Reactivity Hazard:NO

#### **REGULATORY LISTS SEARCHED:**

01-1=IARC Group 1	03=EPCRA 313
01-2A=IARC Group 2A	04=CA Proposition 65
01-2B=IARC Group 2B	05=MA RTK

02=NTP Carcinogen 06=NJ RTK 07=PA RTK

No components of this material were found on the regulatory lists above.

#### CHEMICAL INVENTORIES:

All components comply with the following chemical inventory requirements: EINECS (European Union), IECSC (China), KECI (Korea), PICCS (Philippines), TSCA (United States).

One or more components has been notified but may not be listed in the following chemical inventories: DSL (Canada). Secondary notification by the importer may be required.

One or more components does not comply with the following chemical inventory requirements: AICS (Australia), ENCS (Japan).

#### **NEW JERSEY RTK CLASSIFICATION:**

Under the New Jersey Right-to-Know Act L. 1983 Chapter 315 N.J.S.A. 34:5A-1 et. seq., the product is to be identified as follows: PETROLEUM OIL (Motor oil)

#### WHMIS CLASSIFICATION:

This product is not considered a controlled product according to the criteria of the Canadian Controlled Products Regulations.

SECTION 16 OTHER INFORMATIC	

**NFPA RATINGS:** Health: 0 Flammability: 1 Reactivity: 0

HMIS RATINGS: Health: 1 Flammability: 1 Reactivity: 0

(0-Least, 1-Slight, 2-Moderate, 3-High, 4-Extreme, PPE:- Personal Protection Equipment Index recommendation, \*- Chronic Effect Indicator). These values are obtained using the guidelines or published evaluations prepared by the National Fire Protection Association (NFPA) or the National Paint and Coating Association (for HMIS ratings).

#### LABEL RECOMMENDATION:

Label Category : ENGINE OIL 1 - ENG1

**REVISION STATEMENT:** This is a new Material Safety Data Sheet. **Revision Date:** October 02, 2006

#### ABBREVIATIONS THAT MAY HAVE BEEN USED IN THIS DOCUMENT:

TLV - Threshold Limit Value	TWA - Time Weighted Average
STEL - Short-term Exposure Limit	PEL - Permissible Exposure Limit
	CAS - Chemical Abstract Service Number
ACGIH - American Conference of Government	IMO/IMDG - International Maritime Dangerous Goods
Industrial Hygienists	Code
API - American Petroleum Institute	MSDS - Material Safety Data Sheet
CVX - Chevron	NFPA - National Fire Protection Association (USA)
DOT - Department of Transportation (USA)	NTP - National Toxicology Program (USA)
IARC - International Agency for Research on	OSHA - Occupational Safety and Health Administration
Cancer	

Prepared according to the OSHA Hazard Communication Standard (29 CFR 1910.1200) and the ANSI MSDS Standard (Z400.1) by the Chevron Energy Technology Company, 100 Chevron Way, Richmond, California 94802.

The above information is based on the data of which we are aware and is believed to be correct as of the date hereof. Since this information may be applied under conditions beyond our control and with which we may be unfamiliar and since data made available subsequent to the date hereof may suggest modifications of the information, we do not assume any responsibility for the results of its use. This information is furnished upon condition that the person receiving it shall make his own determination of the suitability of the material for his particular purpose.



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 1. PRODUCT AND COMPANY IDENTIFICATION

Product Name	Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)
Other Trade Names	ABC, Ammonium Phosphate, Monoammonium Phosphate, Tri-Class
Manufacturer/Supplier	Kidde – Residential and Commercial A United Technologies Company
Address	1016 Corporate Park Drive Mebane, NC 27302 USA
Phone Number	(919) 304-8200 (919) 563-5911
Chemtrec Number	(800) 424-9300
(for emergencies only)	(703) 527-3887 (International)
Revision Date:	August 7, 2007
MSDS Date:	January 15, 2007

This MSDS has been compiled in accordance with - EC Directive 91/155/EC - OSHA's Hazcom Standard (29 CFR 1910.1200)

# 2. COMPOSITION/INFORMATION ON THE COMPONENTS

<b>Component Name</b> Monoammonium Phosphate	<b>CAS#/Codes</b> 7722-76-1 EC#2317645	<b>Concentration</b> 85 - 97%	<b>R Phrases</b> None	EU Classification None
Ammonium Sulfate	7783-20-2 EC#2319841	1-6%	None	None
Mica	12001-26-2	1 - 4%	None	None
Clay	8031-18-3	<2%	None	None
Amorphous Silica	7631-86-9 EC#2315454	<2%	None	None
Dye	NA	<0.1%	None	None

# 3. HAZARD IDENTIFICATION

EU Main Hazards Non Hazardous Powder	
Routes of Entry - Eye contact - Inhalation - Skin contact	
Carcinogenic Status See Section 11 - Toxicity	
<b>Target Organs</b> - Respiratory System - Skin <i>-</i> Eye	
Health Effects - Eyes Contact for short periods of time may cause irritation.	
Health Effects - Skin Contact may cause mild irritation.	



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 3. HAZARD IDENTIFICATION

## Health Effects - Ingestion

Ingestion is not an expected route of exposure.

#### **Health Effects - Inhalation**

May irritate the respiratory tract. May cause transient cough and shortness of breath.

# 4. FIRST AID MEASURES

#### Eyes

Immediately flood the eye with plenty of water of warm water for at least 15 minutes, holding the eye open. Obtain medical attention if soreness or redness persists.

#### Skin

Wash affected area with soap and water. Obtain medical attention if irritation persists.

#### Ingestion

Dilute by drinking large quantities of water and obtain medical attention.

#### Inhalation

Move victim to fresh air. Obtain medical attention immediately for any breathing difficulty.

#### Advice to Physicians

Treat symptomatically.

# 5. FIRE FIGHTING MEASURES

## Extinguishing Media

This preparation is used as an extinguishing agent and therefore is not a problem when trying to control a blaze. Use extinguishing agent appropriate to other materials involved. Keep pressurized extinguishers and surroundings cool with water spray as they may rupture or burst in the heat of a fire.

## Unusual Fire and Explosion Hazards

Pressurized containers may explode in heat of fire.

## Protective Equipment for Fire-Fighting

Wear full protective clothing and self-contained breathing apparatus as appropriate for specific fire conditions.

# 6. ACCIDENTAL RELEASE MEASURES

Sweep up or vacuum. Prevent skin and eye contact. Wear appropriate protective equipment.

# 7. HANDLING AND STORAGE

Pressurized extinguishers should be properly stored and secured to prevent falling or being knocked over. Do not drag, slide or roll extinguishers. Do not drop extinguishers or permit them to strike against each other. Never apply flame or localized heat directly to any part of the extinguisher or plastic container. Store pressurized extinguishers and plastic containers away from high heat sources. Storage area should be: - cool - dry - well ventilated - under cover - out of direct sunlight

Revision Date: August 7, 2007



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 8. EXPOSURE CONTROLS/PERSONAL PROTECTION

#### **Occupational Exposure Standards**

Occupational exposure limits are listed below, if they exist. Mica ACGIH TLV: 3 mg/m3 TWA, measured as respirable fraction of the aerosol. OSHA PEL: 20 mppcf, <1% crystalline silica Nuisance Dust Limit OSHA PEL: 50 mppcf or 15 mg/m3 TWA, total dust 15 mppcf or 5 mg/m3 TWA, respirable fraction

## Engineering Control Measures

Use with adequate ventilation. There should be local procedures for the selection, training, inspection and maintenance of this equipment. When used in large volumes, use local exhaust ventilation.

#### **Respiratory Protection**

Not normally required. Use dust mask where dustiness is prevalent, or TLV is exceeded.

#### Hand Protection

Not normally needed when used as a portable fire extinguisher. Use gloves if irritation occurs.

#### **Eye Protection**

Chemical goggles or safety glasses with side shields.

## **Body Protection**

Normal work wear.

# 9. PHYSICAL AND CHEMICAL PROPERTIES

- Physical State Color Odor Specific Gravity Boiling Range/Point (°C/F) Flash Point (PMCC) (°C/F) Solubility in Water Vapor Density (Air = 1) Vapor Pressure Evaporation Rate
- Powder Pale Yellow Odorless Not available Not applicable Not Flammable Not applicable Heavier than air. Not applicable

# 10. STABILITY AND REACTIVITY

#### Stability

Stable under normal conditions. Conditions to Avoid - Heat - High temperatures - Exposure to direct sunlight Materials to Avoid

- Strong oxidizing agents - strong acids - sodium hypochlorite



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 10. STABILITY AND REACTIVITY

## Hazardous Polymerization

Will not occur.

#### **Hazardous Decomposition Products**

- oxides of carbon - ammonia - oxides of phosphorus - nitrogen oxides

# 11. TOXICOLOGICAL INFORMATION

## **Acute Toxicity**

Low order of acute toxicity.

#### **Chronic Toxicity/Carcinogenicity**

This product is not expected to cause long term adverse health effects.

Mica and clay may contain small quantities of quartz (crystalline silica) as an impurity. Prolonged exposure to respirable crystalline silica dust at concentrations exceeding the occupational exposure limits may increase the risk of developing a disabling lung disease known as silicosis. IARC found limited evidence for pulmonary carcinogenicity of crystalline silica in humans.

#### Genotoxicity

This product is not expected to cause any mutagenic effects.

#### Reproductive/Developmental Toxicity

This product is not expected to cause adverse reproductive effects.

# 12. ECOLOGICAL INFORMATION

#### Mobility

No relevant studies identified.

#### Persistence/Degradability

No relevant studies identified.

#### **Bio-accumulation**

No relevant studies identified. Ecotoxicity No relevant studies identified.

## 13. DISPOSAL

Dispose of container in accordance with all applicable local and national regulations. Do not cut, puncture or weld on or near to the container. No harm to the environment is expected from this preparation.

regulated regulated

# 14. TRANSPORT INFORMATION

DOT CFR 172.101 Data	Not re
UN Proper Shipping Name	Not re
UN Class	None
UN Number	None
UN Packaging Group	None



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 15. REGULATORY INFORMATION

## EU Label Information

Classification and labelling have been performed according to EU directives 67/548/EEC and 99/45/EC including amendments.

## EU Hazard Symbol and Indication of Danger.

This preparation is not classified as dangerous.

R phrases

None

S phrases

None.

## US REGULATIONS (Federal, State) and INTERNATIONAL CHEMICAL REGISTRATION LAWS

#### TSCA Listing

This product contains ingredients that are listed on or exempt from listing on the EPA Toxic Substance Control Act Chemical Substance Inventory.

#### **EINECS** Listing

All ingredients in this product have not been verified for listing on the European Inventory of Existing Commercial Chemical Substances (EINECS) or the European List of New Chemical Substances (ELINCS).

## DSL/NDSL (Canadian) Listing

All ingredients in this product are listed on the Domestic Substance List (DSL) or the Non-Domestic Substance List (NDSL) or are exempt from listing.

## WHMIS Classification

D2B

This product was classified in accordance with the hazard criteria of the Canadian Controlled Products Regulations and the MSDS contains all the information required by these regulations.

#### MA Right To Know Law

All components have been checked for inclusion on the Massachusetts Substance List (MSL). Those components present at or above the de minimus concentration include: - Mica (12001-26-2) 1-4% - Amorphous Silica (7631-86-9) <2% - Ammonium Sulfate (7783-20-2) 1-6%

## PA Right To Know Law

This product contains the following chemicals found on the Pennsylvania Hazardous Substance List: -Mica (12001-26-2) 1-4% - Amorphous Silica (7631-86-9) <2% - Ammonium Sulfate (7783-20-2) 1-6%

## NJ Right To Know Law

This product contains the following chemicals found on the NJ Right To Know Hazardous Substance List: - Mica (12001-26-2) 1-4% - Amorphous Silica (7631-86-9) <2%

#### **California Proposition 65**

This product does not contain materials which the State of California has found to cause cancer, birth defects or other reproductive harm.

## SARA Title III Sect. 302 (EHS)

This product does not contain any chemicals subject to SARA Title III Section 302.

## SARA Title III Sect. 304

This product does not contain any chemicals subject to SARA Title III Section 304.



Kidde 90 Multi-Purpose ABC Dry Chemical (Fire Extinguishing Agent)

# 15. REGULATORY INFORMATION

## SARA Title III Sect. 311/312 Categorization

- Immediate (Acute) Health Hazard

SARA Title III Sect. 313 This product does not contain any chemicals that are listed in Section 313 at or above de minimis concentrations.

# 16. OTHER INFORMATION

## NFPA Ratings

NFPA Code for Health - 1 NFPA Code for Flammability - 0 NFPA Code for Reactivity - 0 NFPA Code for Special Hazards - None

#### **HMIS Ratings**

HMIS Code for Health - 1 HMIS Code for Flammability - 0 HMIS Code for Reactivity - 0 HMIS Code for Personal Protection - See Section 8

## Abbreviations

N/A: Denotes no applicable information found or available CAS#: Chemical Abstracts Service Number ACGIH: American Conference of Governmental Industrial Hygienists OSHA: Occupational Safety and Health Administration TLV: Threshold Limit Value PEL: Permissible Exposure Limit STEL: Short Term Exposure Limit NTP: National Toxicology Program IARC: International Agency for Research on Cancer R: Risk S: Safety **Prepared By:** The information contained herein is based on data believed to

The information contained herein is based on data believed to be accurate. However, no representation, warranty, or guarantee is made to its accuracy, reliability or completeness. It is the user's responsibility to satisfy himself as to the suitability and completeness of such information for its own particular use. Kidde – Residential and Commercial assumes no responsibility for personal injury or property damage resulting from use, handling or from contact with this product.

MSDS Number: <b>H3880</b> * * * * * <i>Effective Date:</i> 01/19/06 * * * * * Supercedes: 09/24/04	cause deep ulcers and discolor skin. <b>Eye Contact:</b> Corrostve! Vapors are irritating and may cause damage to the eyes. Contact may	for other purposes. When diluting, the acid should always be added slowly to water and in small amounts. Never use hot water and never add water to the acid. Water added to acid can cause uncontrolled boiling and splashing. When opening
MSDS Material Safety Data Sheet	cause severe burns and permanent eye damage. Chronic Exposure: Long-term exposure to concentrated vapors may cause erosion of teeth. Long term exposures seldonn occur due to the corrosive properties of the acid. Aggravation of Pre-existing Conditions:	metal containers, use non-sparking tools because of the possibility of hydrogen gas being present. Containers of this material may be hazardous when empty since they retain product residues (vapors, liquid); observe all warnings and precautions listed for the product.
From Malinacreationates inter INT Malinckroads Linder 222 Red Short Lane NT CHEMICAIS Linder Process Construction contract and another memory of the contract and another memory and the another process contract another anot	Persons with pre-existing skin disorders or eye disease may be more susceptible to the effects of this substance.	8. Exposure Controls/Personal Protection
All new entropiercy constructs through the descript is Constructe. Service 1: 860-682 (55.27) for antisknews	4. First Aid Measures	Airborne Exposure Limits: For Hydrochloric acid:
HYDROCHLORIC ACID, 33 - 40%	Inhalation: Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult rows or wear. Ger medical attention immediately.	<ul> <li>OSHA Permissible Exposure Limit (PEL):</li> <li>5 ppm (Ceiling)</li> <li>ACGH Threshold Limit Value (TLV):</li> <li>7 mm (Ceiling) 4A Nor classifiable as a hinman carcinoren</li> </ul>
1. Product Identification	Ingestion: DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical	Veritiation System: A system of local and/or general exhaust is recommended to keep employee exposures below the Airbonne Exposure Limits. Local exhaust ventilation is
Synonyms: Murtatic acid; hydrogen chloride, aqueous CAS No.: 7647-01-0 MAAnonity Wighty 54.44	attention immediatety. Skin Contact: In case of contact, immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before	generary preterior or excaves to control true contrastrum or the contraminant or the source, preventing dispersion of it into the general work area. Please refer to the ACIH document, <i>Industrial Ventilation</i> , <i>A Manual of Recommended Practices</i> , most recent edition, for details.
moneculari weigut. 50.40 Chemical Formula: HCl Product Codes: J.T. Baker: 5367, 5575, 5800, 5814, 5821, 5839, 5861, 5862, 5894, 5962, J.T. Baker: 5567, 5537, 5575, 5800, 5814, 5821, 5530, 5536, 9538, 9530, 9540,	reuse. Thoroughly clean shoes before reuse. Get medical attention immediately. <b>Eye Contact:</b> Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.	Personal Respirators (NIOSH Approved): If the exposure limit is exceeded, a full facepiece respirator with an acid gas cartridge may be worn up to 50 times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier.
9544, 0548 Mallinckrodi: 2062. 2515, 2612, 2624. 2626. 3861, 5583, 5587, H611, H613, H987, H992, H999, V078, V628	S. Fire Fighting Measures	whichever is lowest. For emergencies or instances where the exposure levels are not known, use a full-facepicce positive-pressure, air-supplied respirator. WARNING: Air purifying respirators do not protect workers in oxygen-deficient atmoscheres.
2. Composition/Information on Ingredients	Fire: Extreme heat or contact with metals can release flammable hydrogen gas.	Skin Protection: Rubber or neoprene gloves and additional protection including impervious boots, apron. or coveralls, as needed in areas of unusual exposure to prevent skin contact. Ex.o. Provensional
Percent 33	<b>Explosion:</b> Not considered to be an explosion hazard. <b>Fire Extinguishing Media:</b>	by troction. Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.
7732-18-5 60 - 67%	If involved in a fire, use water spray. Neutralize with soda ash or slaked lime. Special Information: In the event of a fire, wear full protective clothing and NIOSH-approved self-	9. Physical and Chemical Properties
3. Hazards Identification	contained breathing apparatus with full facepicce operated in the pressure demand or other positive pressure mode. Structural firefighter's protective clothing is ineffective for fires involving hydrochloric acid. Stay away from ends of tanks. Cool tanks with water spray until well after fire is out.	Appearance: Colorless, fuming liquid. Odor
Emergency Overview POISON: DANGER: CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE. MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY CAUSE LUNG DAMAGE.	6. Accidental Release Measures	Pungent odor of hydrogen chloride. Solubility: Infinite in water with slight evolution of heat. Density:
<ul> <li>SAF-T-DATA<sup>(m)</sup> Ratings (Provided here for your convenience) Health Rating: 3 - Severe (Poison) Flammability Rating: 0 - None Reactivity Rating: 2 - Moderate Contact Rating: 4 - Extreme (Corrosive) Lab Protective Equip: GOGGLES &amp; SHIELD; LAB COAT &amp; APRON: VENT HOOD: PROPER GLOVES</li> <li>Storage Color Code: White (Corrosive)</li> </ul>	Ventilate area of leak or spill. Wear appropriate personal protective equipment as specified in Section 8. Isolate hazard area. Keep unnecessary and unprotected personnel from entering. Contain and recover liquid when possible. Neutralize with alkaline material (soda ash. lime), then absorb with an inert material (e. g., vermiculite, dry sand, earth), and place in a chemical waste container. Do not use combinishie materials, such as saw dust. Do not flush to sewer! US Regulations (CERCLA) require reporting spills and releases to soil, water and air in excess of reportable quantities. The toil free number for the US Coast Guard National	1.18 pH: For HCL solutions: 0.1 (1.0 N), 1.1 (0.1 N), 2.02 (0.01 N) % Volatiles by volume @ 21C (70F): 100 Builing Point: 53C (127F) Azeotrope (20.2%) boils at 109C (228F) Melting Point: -74C (-101F)
Potential Health Effects Inhalation: Corrosive! Inhalation of vapors can cause coughing, choking, inflammation of the nose, throat, and unsor resorratory tract, and in severe cases, pulmonary edema.	Kesponse Center is (800) 4.24-8802. J. T. Baker NEUTRASORB® acid neutralizers are recommended for spills of this product.	Vapor Density (Air=1): No information found. Vapor Pressure (mm Hg): 1900 @25C (77F) Fvon @25C (77F)
circulatory failure, and death. Tagestion:	7. Handling and Storage	No information found.
Construction of the anticomment of the account of the model of the model, throad, esplaying and gastrointestinal tract. May cause nausea, vomiting, and diarrhea. Swallowing may be fatal. Skin Contact: Corrosive! Can cause redness, pain, and severe skin burns. Concentrated solutions	Store in a cool, dry, ventilated storage area with acid resistant floors and good drainage. Protect from physical damage. Keep out of direct sunlight and away from heat, water, and incompatible materials. Do not wash out container and use it	10. Stability and Reactivity

ua, JUJ.1.) ing Namne: HYDROCHLORIC ACID : 8 789	Point and the intervent of the second
reported for product/size: 475LB (Water, 1.M.O.) ing Name: HYDROCHLORIC ACID :: 8	16. Other Information
789 p. 11 reported for product/size: 475LB	NFPA Ratings: Health. 3 Flammability: 0 Reactivity: 1 Label Hazard Warning: POISON: DANGER! CORROSIVE. LIQUID AND MIST CAUSE SEVERE BURNS TO ALL BODY TISSUE: MAY BE FATAL IF SWALLOWED OR INHALED. INHALATION MAY
y Information	CADDE LUNG DAMALE. Label FLOWED DAMALE. Do not get in eves, on skin, or on clothing. Do not breathe vapor or mist. Use only with adequate variatiation. Wath throroughly after handling promptly. Store in a tightive closed container Remove and wash containmated clothing promptly.
y Phrases	Label First Aid: In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If swallowed, DO
26-45	NOT INDUCE VOMITING. Give large quantities of water. Never a pive anything by mouth to an unconscious person. If inhaled, remove to fresh air. If noh breathing, give artificial respiration. If breathing is difficult give oxyger. In all cases ger medical attention immediately.
Inventory Status - Part 1/ Tt TSCA EC Appan Australia Chloride (7647-01-0) Yes	Product Use: Luboratory: Reagent Revision Information: MSDS Section(s): charged since last revision of document include: 16. Distalamers:
Inventory Status - Part 2/	Mallineterodi Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is
nternational TPQ Lis (7647-01-0)	menued only as a gute to ne appropriate presentionary juantuming on measure up a properly trained presenting the product. Individuals receiving the information must service their independent judgment in determining its appropriateness for a particular purpose. MALLINGERODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRAVITES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT WARRAVITES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT DATE WARRAVITES, EITHER EXPRESS OR IMPLIED, INCLUDING WARRAVITES, EITHER EXPRESS OR IMPLIED, EITHER EXPRESS OR IMPLIED, EITHER EXPRESS OR IMPLIED, EI
International Regulations -	LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNES FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREN OR THE PRODUCT TO WHICH THE INFORMATION REFERS.
t CERCLA 261.33 8(d) Chloride (7647-01-0) 5000 No 32-18-5) No No No	ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION.
ns Convention: N	Prepared by: Environmental Health & Safety Pronorman Number (24) 634-1600 (U.S.A.)
<u>Hydrochloric Acid Overview</u>	
<u>Health Risk:3 - Severe (Poison)</u> <u>Flammability:0 - None</u> <u>Reactivity Rating:2 - Moderate</u> Contact Rating:4 - Extreme (Corrosive)	
<u>ive Equipment Required:</u> Proper Gloves, Goggles	
ush skin with water for 15 minutes. Remove contaminated clothing and shoes.	ted clothing and shoes. edical attention immediatelv.

First Aid Measures: Immediately flush skin with water for 15 minute.

Protective Equipment Required: Proper

Immediately flush eyes with plenty of water for 15 minutes, lifting lower and upper eyelids. Get medical attention immediately.

Incompatibilities: Will not occur

A strong mineral acid, concentrated hydrochloric acid is incompatible with many substances and highly reactive with strong bases, metals, metal oxides, hydroxides, amines, carbonates and other alkaline materials. Incompatible with materials such as evandes, sulfides, audites, and formaldehy de.

Conditions to Avoid: Heat, direct sunlight

# 11. Toxicological Information

Inhalation rat LC50: 3124 ppm/1H; oral rabbit LD50: 900 mg/kg (Hydrochloric acid concentrated); investigated as a tumorigen, mutagen, reproductive effector. IARC Category None o No No Anticipated 1-0) No No N Hydrogen Chloride (7647-01-0) Water (7732-18-5) Cancer Lists/Carcinogen---Ingredient Known A

# 12. Ecological Information

Environmental Fate: When released into the soil, this material is not expected to biodegrade. When released into the soil, this material may leach into groundwater. Environmental Toxicity: This material is expected to be toxic to aquatic life

# 13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved waste facility. Processing, use or communation of this product may change the waste management options. State and local disposal regulations may differ from defactal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

14. Transport Information

Proper Shipping Name: HYDROCHLORIC ACID Hazard Class: 8 UN/NA: UN1789 Packing Group: II Information reported for product/size: 475LB Packing Group: II Information reported for product/size: 475LB International (Water, I.M.O.)

Proper Shipping Name: HYDROCHLORIC ACID Hazard Class: 8

UN/NA: UN1789

Hzzardous Decomposition Products: When heated to decomposition, emits toxic hydrogen chloride fumes and will react with water or steam to produce heat and toxic and corrosive fumes. Thermal oxidative decomposition

produces toxic chlorine fumes and explosive hydrogen gas.

Hazardous Polymerization:

Stable under ordinary conditions of use and storage. Containers may burst when heated

Stability:

Domestic (Land, D.O.T.)

Australian Hazchem Code: 2R

# 15. Regulatory Information

Risk and Safety Phrases: Symbol: C Risk: 34-37 Safety: (1/2-)26-45 Chemical Inventory Status - Part 1/--------Ingredient TSCA EC Japan Australia Hydrogen Choride (7647-01-0) Yes Yes Yes Water (7732-18-5)

0 0 N N NDSL Chemical Inventory Status - Part 2/-----Ingredient Korea DSL NU Hydrogen Chloride (7647-01-0) Yes Yes Water (7732-18-5) Yes Yes Federal, State & International Regulations - Fa Ingredient RQ TPQ List Chemical Card Hydrogen Chloride (7417-01-0) 5000 500+ Ye Water (7732-18-5) No No No

Federal, State & International Regulations - Pa -TSCA-

8 (d) ŶZ Ő 261.33 5000 b No Jurrent CERCLA Hydrogen Chloride (7647-01-0) Water (7732-18-5)

TSCA 12(b) No Chemical Weapons Convention: Yes SARA 311/312: Acute: Yes

Chronic: Yes Fi QN

(Mixture / Liquid) Reactivity: No

# MSDS Number: **S4034** \* \* *Effective Date:* 05/04/07 \* \* \* *Supercedes:* 07/07/04



SODIUM HYDROXIDE

#### 1. Product Identification

Synonyms: Caustic soda; Iye; sodium hydroxide solid; sodium hydrate CAS No.: 1310-73-2 Molecular Weight: 40.00 Chemical Formula: NaOH Product Codes: J.T. Baker: 1508, 3717, 3718, 3721, 3722, 3723, 3728, 3734, 3736, 5045, 5565 Mallinckrodt: 7001, 7680, 7708, 7712, 7772, 7798

#### 2. Composition/Information on Ingredients

Ingredient	CAS No	Percent	
Hazardous			
Sodium Hydroxide	1310-73-2	99 - 100%	Yes

#### 3. Hazards Identification

#### **Emergency Overview**

#### POISON! DANGER! CORROSIVE. MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES BURNS TO ANY AREA OF CONTACT. REACTS WITH WATER, ACIDS AND OTHER MATERIALS.

SAF-T-DATA<sup>(tm)</sup> Ratings (Provided here for your convenience)

Health Rating: 4 - Extreme (Poison) Flammability Rating: 0 - None Reactivity Rating: 2 - Moderate Contact Rating: 4 - Extreme (Corrosive) Lab Protective Equip: GOGGLES & SHIELD; LAB COAT & APRON; VENT HOOD; PROPER GLOVES Storage Color Code: White Stripe (Store Separately)

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#### Potential Health Effects

#### Inhalation:

Severe irritant. Effects from inhalation of dust or mist vary from mild irritation to serious damage of the upper respiratory tract, depending on severity of exposure. Symptoms may include sneezing, sore throat or runny nose. Severe pneumonitis may occur.

#### Ingestion:

Corrosive! Swallowing may cause severe burns of mouth, throat, and stomach. Severe scarring of tissue and death may result. Symptoms may include bleeding, vomiting, diarrhea, fall in blood pressure. Damage may appear days after exposure.

#### Skin Contact:

Corrosive! Contact with skin can cause irritation or severe burns and scarring with greater exposures.

#### Eye Contact:

Corrosive! Causes irritation of eyes, and with greater exposures it can cause burns that may result in permanent impairment of vision, even blindness.

#### Chronic Exposure:

Prolonged contact with dilute solutions or dust has a destructive effect upon tissue. Aggravation of Pre-existing Conditions:

Persons with pre-existing skin disorders or eye problems or impaired respiratory function may be more susceptible to the effects of the substance.

#### 4. First Aid Measures

#### Inhalation:

Remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

#### Ingestion:

DO NOT INDUCE VOMITING! Give large quantities of water or milk if available. Never give anything by mouth to an unconscious person. Get medical attention immediately.

#### Skin Contact:

Immediately flush skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Call a physician, immediately. Wash clothing before reuse.

#### Eye Contact:

Immediately flush eyes with plenty of water for at least 15 minutes, lifting lower and upper eyelids occasionally. Get medical attention immediately.

#### Note to Physician:

Perform endoscopy in all cases of suspected sodium hydroxide ingestion. In cases of severe esophageal corrosion, the use of therapeutic doses of steroids should be considered. General supportive measures with continual monitoring of gas exchange, acid-base balance, electrolytes, and fluid intake are also required.

#### 5. Fire Fighting Measures

#### Fire:

Not considered to be a fire hazard. Hot or molten material can react violently with water. Can react with certain metals, such as aluminum, to generate flammable hydrogen gas.

#### Explosion:

Not considered to be an explosion hazard.

#### Fire Extinguishing Media:

Use any means suitable for extinguishing surrounding fire. Adding water to caustic solution generates large amounts of heat.

#### Special Information:

In the event of a fire, wear full protective clothing and NIOSH-approved selfcontained breathing apparatus with full facepiece operated in the pressure demand or other positive pressure mode.

#### 6. Accidental Release Measures

Ventilate area of leak or spill. Keep unnecessary and unprotected people away from area of spill. Wear appropriate personal protective equipment as specified in Section 8. Spills: Pick up and place in a suitable container for reclamation or disposal, using a method that does not generate dust. Do not flush caustic residues to the sewer. Residues from spills can be diluted with water, neutralized with dilute acid such as acetic, hydrochloric or sulfuric. Absorb neutralized caustic residue on clay, vermiculite or other inert substance and package in a suitable container for disposal.

US Regulations (CERCLA) require reporting spills and releases to soil, water and

air in excess of reportable quantities. The toll free number for the US Coast Guard National Response Center is (800) 424-8802.

#### 7. Handling and Storage

Keep in a tightly closed container. Protect from physical damage. Store in a cool, dry, ventilated area away from sources of heat, moisture and incompatibilities. Always add the caustic to water while stirring; never the reverse. Containers of this material may be hazardous when empty since they retain product residues (dust, solids); observe all warnings and precautions listed for the product. Do not store with aluminum or magnesium. Do not mix with acids or organic materials.

#### 8. Exposure Controls/Personal Protection

#### Airborne Exposure Limits:

- OSHA Permissible Exposure Limit (PEL):

2 mg/m3 Ceiling

- ACGIH Threshold Limit Value (TLV):

2 mg/m3 Ceiling

#### Ventilation System:

A system of local and/or general exhaust is recommended to keep employee exposures below the Airborne Exposure Limits. Local exhaust ventilation is generally preferred because it can control the emissions of the contaminant at its source, preventing dispersion of it into the general work area. Please refer to the ACGIH document, *Industrial Ventilation, A Manual of Recommended Practices*, most recent edition, for details.

#### Personal Respirators (NIOSH Approved):

If the exposure limit is exceeded and engineering controls are not feasible, a half facepiece particulate respirator (NIOSH type N95 or better filters) may be worn for up to ten times the exposure limit or the maximum use concentration specified by the appropriate regulatory agency or respirator supplier, whichever is lowest. A full-face piece particulate respirator (NIOSH type N100 filters) may be worn up to 50 times the exposure limit, or the maximum use concentration specified by the appropriate regulatory agency, or respirator supplier, whichever is lowest. If oil particles (e.g. lubricants, cutting fluids, glycerine, etc.) are present, use a NIOSH type R or P filter. For emergencies or instances where the exposure levels are not known, use a full-facepiece positivepressure, air-supplied respirator. WARNING: Air-purifying respirators do not protect workers in oxygen-deficient atmospheres.

#### Skin Protection:

Wear impervious protective clothing, including boots, gloves, lab coat, apron or coveralls, as appropriate, to prevent skin contact.

#### Eye Protection:

Use chemical safety goggles and/or a full face shield where splashing is possible. Maintain eye wash fountain and quick-drench facilities in work area.

#### 9. Physical and Chemical Properties

#### Appearance:

White, deliquescent pellets or flakes. Odor: Odorless. Solubility: 111 g/100 g of water. Specific Gravity: 2.13 pH: 13 - 14 (0.5% soln.) % Volatiles by volume @ 21C (70F): 0

Boiling Point:
1390C (2534F)
Melting Point:
318C (604F)
Vapor Density (Air=1):
> 1.0
Vapor Pressure (mm Hg):
Negligible.
Evaporation Rate (BuAc=1):
No information found.

Page 2

#### 10. Stability and Reactivity

#### Stability:

Stable under ordinary conditions of use and storage. Very hygroscopic. Can slowly pick up moisture from air and react with carbon dioxide from air to form sodium carbonate.

#### **Hazardous Decomposition Products:**

Sodium oxide. Decomposition by reaction with certain metals releases flammable and explosive hydrogen gas.

Hazardous Polymerization:

#### Will not occur.

Incompatibilities:

Sodium hydroxide in contact with acids and organic halogen compounds, especially trichloroethylene, may causes violent reactions. Contact with nitromethane and other similar nitro compounds causes formation of shocksensitive salts. Contact with metals such as aluminum, magnesium, tin, and zinc cause formation of flammable hydrogen gas. Sodium hydroxide, even in fairly dilute solution, reacts readily with various sugars to produce carbon monoxide. Precautions should be taken including monitoring the tank atmosphere for carbon monoxide to ensure safety of personnel before vessel entry. **Conditions to Avoid:** 

Moisture, dusting and incompatibles.

#### 11. Toxicological Information

Irritation data: skin, rabbi investigated as a mutager \Cancer Lists	ı.	, <b>,</b>		
Ingredient Category	Known	Anticipated	ł	IARC
Sodium Hydroxide None	(1310-73-	2) No N	No	

#### 12. Ecological Information

**Environmental Fate:** No information found. **Environmental Toxicity:** No information found.

#### 13. Disposal Considerations

Whatever cannot be saved for recovery or recycling should be handled as hazardous waste and sent to a RCRA approved waste facility. Processing, use or contamination of this product may change the waste management options. State and local disposal regulations may differ from federal disposal regulations. Dispose of container and unused contents in accordance with federal, state and local requirements.

#### 14. Transport Information

#### Domestic (Land, D.O.T.)

Proper Shipping Name: SODIUM HYDROXIDE, SOLID Hazard Class: 8 UN/NA: UN1823 Packing Group: II Information reported for product/size: 300LB

#### International (Water, I.M.O.)

Proper Shipping Name: SODIUM HYDROXIDE, SOLID Hazard Class: 8 UN/NA: UN1823 Packing Group: II Information reported for product/size: 300LB

#### 15. Regulatory Information

Chemical Inventory Status - Part 1\	
Ingredient TSCA EC Japan Australia	
Sodium Hydroxide (1310-73-2) Yes Yes Yes	Yes
Chemical Inventory Status - Part 2\	
Ingredient Korea DSL NDSL Phi.	1.
Sodium Hydroxide (1310-73-2) Yes Yes No	Yes
Federal, State & International Regulations - Part	1\
-SARA 302SARA 313	
Ingredient RQ TPQ List Chemical Catg	
Sodium Hydroxide (1310-73-2) No No No	No
Federal, State & International Regulations - Part	2\
-RCRATSCA-	

Ingredient	CERCLA	261	.33	8(d)	
Sodium Hydroxide	(1310-73-2)	1000	No		No
Chemical Weapons	Convention:	No	TSCA 12	(b):	No
CDTA: No					
SARA 311/312: A	cute: Yes	Chroni	.c: No	Fire	No
Pressure: No					
Reactivity: Yes	(Pure	/ Solid	1)		

#### Sodium Hydroxide Overview

<u>Health Risk:3 – Severe (Poison)</u> <u>Flammability:0 - None</u> <u>Reactivity Rating:2 – Moderate</u> <u>Contact Rating:4 – Extreme (Corrosive)</u>

#### Australian Hazchem Code: 2R Poison Schedule: S6 WHMIS: This MSDS has been prepared according to the hazard criteria of the Controlled Products Regulations (CPR) and the MSDS contains all of the information required by the CPR.

#### 16. Other Information

NFPA Ratings: Health: 3 Flammability: 0 Reactivity: 1 Label Hazard Warning: POISON! DANGER! CORROSIVE, MAY BE FATAL IF SWALLOWED. HARMFUL IF INHALED. CAUSES BURNS TO ANY AREA OF CONTACT. REACTS WITH WATER, ACIDS AND OTHER MATERIALS. Label Precautions: Do not get in eves, on skin, or on clothing. Do not breathe dust. Keep container closed. Use only with adequate ventilation. Wash thoroughly after handling. Label First Aid: If swallowed, DO NOT INDUCE VOMITING. Give large quantities of water. Never give anything by mouth to an unconscious person. In case of contact, immediately flush eyes or skin with plenty of water for at least 15 minutes while removing contaminated clothing and shoes. Wash clothing before reuse. If inhaled, remove to fresh air. If not breathing give artificial respiration. If breathing is difficult, give oxygen. In all cases get medical attention immediately. **Product Use:** Laboratory Reagent. **Revision Information:** No Changes. Disclaimer: \*\*\*\*\*\*\* \*\*\*\*\*\* Mallinckrodt Baker, Inc. provides the information contained herein in good faith but makes no representation as to its comprehensiveness or accuracy. This document is intended only as a guide to the appropriate precautionary handling of the material by a properly trained person using this product. Individuals receiving the information must exercise their independent judgment in determining its appropriateness for a particular purpose. MALLINCKRODT BAKER, INC. MAKES NO REPRESENTATIONS OR WARRANTIES, EITHER EXPRESS OR IMPLIED, INCLUDING WITHOUT LIMITATION ANY WARRANTIES OF MERCHANTABILITY, FITNESS FOR A PARTICULAR PURPOSE WITH RESPECT TO THE INFORMATION SET FORTH HEREIN OR THE PRODUCT TO WHICH THE INFORMATION REFERS. ACCORDINGLY, MALLINCKRODT BAKER, INC. WILL NOT BE RESPONSIBLE FOR DAMAGES RESULTING FROM USE OF OR RELIANCE UPON THIS INFORMATION. \*\*\*\*\* Prepared by: Environmental Health & Safety Phone Number: (314) 654-1600 (U.S.A.)

Protective Equipment Required: Proper Gloves, Goggles

<u>First Aid Measures:</u> Immediately flush skin with water for 15 minutes. Remove contaminated clothing and shoes. Immediately flush eyes with plenty of water for 15 minutes, lifting lower and upper eyelids. **Get medical attention immediately.** 

# **ARCADIS**

Appendix G

ARCADIS H&S Procedures

Infrastructure, environment, facilities	ARCADIS HS Procedure Name Hazardous Waste Operations and Emergency	Revision Number 01
Implementation Date	Response ARCADIS HS Procedure No.	Revision Date
7 September 2009	ARC HSFS012	7 September 2009
Author	Page 1 of 14	Approver
Michael Ramer	<b>v</b>	Mike Thomas

## 1. POLICY

All ARCADIS operations shall meet or exceed all applicable federal, state, and local safety and health regulations pertaining to hazardous waste operations and emergency response (HAZWOPER). All HAZWOPER work performed or managed by ARCADIS shall be performed in accordance with the following requirements, which are intended to ensure compliance with 29 CFR 1910.120 (and equivalent state regulations-see Section 5.5). Conformance with this procedure also covers operations associated with RCRA (Resource Conservation and Recovery Act) and CERCLA (Comprehensive Environmental Response Compensation and Liability Act) sites.

## 2. PURPOSE AND SCOPE

## 2.1 Purpose

This procedure sets forth the requirements for ARCADIS operations on a HAZWOPER site. As required by regulation, ARCADIS must develop and implement a written safety and health program for employees involved in hazardous waste and emergency response operations. This procedure, as well as other referenced procedures and documents, satisfies the program requirement for identification, evaluation, and control of health and safety hazards, and provides guidance requirements for emergency response for hazardous waste operations.

## 2.2 Scope

This procedure applies to employees who work at HAZWOPER sites (as defined) where there is the reasonable potential that employees will be exposed to health and safety hazards associated with the site.

HAZWOPER sites include the following:

- Cleanup operations at uncontrolled hazardous waste sites (e.g. Superfund sites);
- Corrective actions involving cleanup operations at sites covered by the RCRA;
- Voluntary cleanup operations recognized by the regulatory agency;
- Operations involving hazardous wastes that are conducted at treatment, storage and disposal facilities (TSDFs); or
- Emergency response operations.

## 3. **DEFINITIONS**

The following definitions are provided to give understanding to the various requirements in this procedure:

Infrastructure, environment, facilities	ARCADIS HS Procedure Name Hazardous Waste Operations and Emergency Response	<u>Revision Number</u> 01
Implementation Date	ARCADIS HS Procedure No.	Revision Date
7 September 2009	ARC HSFS012	7 September 2009
Author	Page 2 of 14	Approver
Michael Ramer		Mike Thomas

- **Clean-up operation** means an operation where hazardous substances are removed, contained, incinerated, neutralized stabilized, cleared-up, or in any other manner processed or handled with the ultimate goal of making the site safer for people or the environment.
- Emergency response or responding to emergencies means a response effort by employees from outside the immediate release area or by other designated responders (i.e., mutual aid groups, local fire departments, etc.) to an occurrence which results, or is likely to result, in an uncontrolled release of a hazardous substance. Responses to incidental releases of hazardous substances where the substance can be absorbed, neutralized, or otherwise controlled at the time of release by employees in the immediate release area, or by maintenance personnel are not considered to be emergency responses within the scope of this procedure. Responses to releases of hazardous substances where there is no potential safety or health hazard (i.e., fire, explosion, or chemical exposure) are not considered to be emergency responses.
- **Exposure or exposed** means that an employee is subjected in the course of employment to a chemical that is a physical or health hazard, and includes potential (e.g. accidental or possible) exposure; subjected is in terms of health hazards includes any route of entry (e.g. inhalation, ingestion, skin contact or absorption).
- **Hazardous substance** means any substance designated or listed under below, exposure to which results or may result in adverse effects on the health or safety of employees:
  - Any substance defined under section 101(14) of CERCLA;
  - Any biologic agent and other disease causing agent which after release into the environment and upon exposure, ingestion, inhalation, or assimilation into any person, either directly from the environment or indirectly by ingestion through food chains, will or may reasonably be anticipated to cause death, disease, behavioral abnormalities, cancer, genetic mutation, physiological malfunctions (including malfunctions in reproduction) or physical deformations in such persons or their offspring;
  - Any substance listed by the U.S. Department of Transportation as hazardous materials under 49 CFR 172.101 and appendices; and
  - Hazardous waste as defined below.
- **Hazardous waste** means A waste or combination of wastes as defined in 40 CFR 261.3, or those substances defined as hazardous wastes in 49 CFR 171.8.
- **Hazardous waste operation** means any operation conducted within the scope of this procedure.
- **Hazardous waste site** or "Site" means any facility or location within the scope of this procedure at which hazardous waste operations take place.
- Health hazard means a chemical, mixture of chemicals or a pathogen for which there is statistically significant evidence based on at least one study conducted in accordance with established scientific principles that acute or chronic health effects may occur in exposed employees. The term "health hazard" includes chemicals which are carcinogens, toxic or

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highly toxic agents, reproductive toxins, irritants, corrosives, sensitizers, hepatotoxins, nephrotoxins, neurotoxins, agents which act on the hematopoietic system and agents which damage the lungs, skin, eyes, or mucous membranes. It also includes stress due to temperature extremes. Further definition of the terms used above can be found in Appendix A to 29 CFR 1910.1200.

- Site Safety Officer (SSO) means the individual located on a hazardous waste site who is
  responsible to ARCADIS and has the authority and knowledge necessary to implement the
  site safety and health plan and verify compliance with applicable safety and health
  requirements.
- Uncontrolled hazardous waste site means an area identified as an uncontrolled hazardous waste site by a governmental body, whether Federal, state, local or other where an accumulation of hazardous substances creates a threat to the health and safety of individuals or the environment or both. Some sites are found on public lands such as those created by former municipal, county or state landfills where illegal or poorly managed waste disposal has taken place. Other sites are found on private property, often belonging to generators or former generators of hazardous substance wastes. Examples of such sites include, but are not limited to, surface impoundments, landfills, dumps, and tank or drum farms. Normal operations at TSD sites are not covered by this definition.

## Definition – Hazardous Waste Operations Site (HazWoper)

For the purposes of identifying a *HazWoper site* and *training* requirements for ARCADIS personnel, the following considerations must be determined and met:

- Does the site meet the definition of HazWoper site; and
- Will ARCADIS personnel be performing HazWoper defined activities?

A HazWoper site must meet all three of the following criteria:

- 1) A site where **HazWoper activities** are conducted. **HazWoper activities** are defined as follows:
  - Corrective actions and Clean-up operations activities required or known by a governmental body, whether Federal, state, local or other involving hazardous substances (including, but not limited to, contaminant removal; treatment; remediation; etc.); or
  - Site investigation activities where the presence or potential presence of hazardous substances exist (activities include, but are not limited to, Intrusive soil activities including soil boring and sampling, trenching, coring, hand augering; Phase I Environmental Site Assessments; Phase II Site Investigations; Building or Geological Surveys; Well Monitoring/Sampling; etc.)
- 2) Where employee **exposure** or the reasonable possibility for employee exposure to safety or health hazards exists. **Exposure** is defined as follows:

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- Exposure or exposed means that in the course of work, an employee is
   "subjected" to, or has the potential to be "subjected", to a hazardous substance that is a physical or health hazard (includes accidental exposure).
- "Subjected", in terms of health hazards, includes any route of entry (e.g. inhalation, ingestion, skin contact and/or absorption) without regard to the use of personal protective equipment.
- 3) Where the presence or potential presence of **hazardous substance** exists.
  - Hazardous substance is any substance to which exposure results, or may result, in adverse effects to the health or safety of employees (including biologic agents, substances listed by the US Department of Transportation as hazardous materials and hazardous waste).

## 4. **RESPONSIBILITIES**

## 4.1 Employees

Employees are required, in general, to recognize potential adverse exposures and notify health and safety staff, follow prescribed hazard reduction protocols and use Personal Protective Equipment during activities with potential for potentially harmful exposures. In addition, employees have the responsibilities to adhere to this HSP and to communicate H&S concerns, issues and questions to their supervisor or their respective Health and Safety resource. Employees are required to participate in the medical monitoring program, including annual audiogram and chemical and client-specific training as applicable based on their job duties.

On project sites, all employees have the responsibility to:

- Use the TRACK process prior to any activity.
- Follow all ARCADIS and client requirements.
- To understand and appropriately utilize the "Stop Work Authority" concept.
- Read and work in accordance with the components of the site-specific HASP.
- Report all unsafe working conditions to the Site Safety Officer (SSO).
- Report all injuries, no matter how minor, to the SSO.

## 4.2 Managers

In planning and preparation of projects affected by HAZWOPER requirements, the project manager and/or task manager must complete the project-specific H&S Stewardship Checklist & Project Hazard Analysis Worksheet. *Note: The project Hazard Analysis Worksheet uses the Hazard Analysis Risk Control (HARC) ranking process (ARCADIS H&S Procedure ARC HSMS002) (see Section 4 of this HASP).* 

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Additional responsibilities of the project manager and task manager are as follows:

- Review all applicable H&S Procedures, and ensure that project activities conform to all requirements.
- Obtain client-specific health and safety information and communicate with the client on health and safety issues.
- Communicate with the Site Safety Officer (SSO) on health and safety issues.
- Allocate resources for correction of identified unsafe work conditions.
- Ensure ARCADIS site workers have all training necessary for the project.
- Report all injuries, illnesses and near-losses to the Client H&S Resource or Project H&S Manager (PHSM), lead incident investigations, and ensure that any recommendations made are implemented.
- Communicating with and appropriately managing subcontractors, ensuring that employees have appropriate training and qualifications, and for ensuring all client H&S requirements are met.
- Involving the appropriate ARCADIS H&S Staff and project client staff, as necessary.
- Ensuring that all subcontractors have been communicated with concerning the minimum H&S requirements for the project.
- Providing adequate resources and budget for personal protective equipment (PPE).

## 4.3 Principals in Charge (PICs)

Have the responsibility to know and follow all applicable ARCADIS and client H&S requirements, for ensuring work is conducted under the H&S policy and for implementing the procedure requirements provided for in this procedure on any project that pose hazards to ARCADIS employees or employees of its subcontractors, clients, and other organizations present in the vicinity of work controlled by ARCADIS. In addition, PICs responsibilities also include determining and communicating any specific client requirements that are applicable.

## 4.4 Corporate H&S Staff

Have the responsibility for:

- Communicating the policy and procedure requirements in this procedure with all offices within ARCADIS – US.
- Ensuring that offices are aware of this procedure.
- Ensuring this procedure is being implemented effectively.
- Provide required training or guidance on approved training options.

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- Providing the necessary suppliers and criteria for selection of H&S equipment.
- Assuring the development and implementation of this procedure.

## 4.5 Project Health and Safety Manager (PHSM)

The PHSM oversees all aspects of the site safety program, and prepares site-specific health and safety guidance documents or addenda to the HASP. The PHSM does not report to the Project Manager, and is separately accountable to the ARCADIS project team for site health and safety. The PHSM acts as the sole contact to regulatory agencies on matters of safety and health. Other responsibilities include:

- Overall authority for health and safety compliance and HASP conformance for the project.
- General health and safety program administration.
- Conducts project health and safety audits as warranted.
- Determines the level of personal protection required.
- Updates equipment or procedures based on information obtained during site operations.
- Establishes air-monitoring parameters based on expected contaminants.
- Assists in injury, illness and near-miss investigations and follow-up.

## 4.6 Site Safety Officer (SSO)

The SSO is key to the on-site health and safety compliance with regulations and conformance with ARCADIS and client requirements. The SSO is responsible for:Reviews and works in accordance with the components of the HASP. Ensures that the HASP is available to and reviewed by all site personnel including subcontractors.

- Ensures that necessary site-specific training is performed (both initial and "tailgate" safety briefings.
- Ensures site visitors have been informed of the hazards related to ARCADIS work, and have signed the Site Visitors Log.
- Ensures that work is performed in a safe manner and has authority to stop work when necessary to protect workers and/or the public.
- Coordinates activities during emergency situations.
- Ensures that all necessary permits and safety information provided by the client is disseminated to other site personnel and is maintained in an organized manner.

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- Communicates with the PM, Client H&S Resource and/or the PHSM on health and safety issues.
- Reports all injuries, illnesses and near-misses to the PM, Client H&S Resource and Project Health and Safety Manager.
- Ensures that necessary safety equipment is maintained and used at the site.
- Contacts a health and safety professional for assistance in establishing the respiratory cartridge change schedule as required.

## 5. PROCEDURE

To comply with regulation and conform to this procedure, the following sections describe the ARCADIS requirements associated with HAZWOPER projects.

## 5.1 General Requirements

The health and safety program to meet the HAZWOPER requirements shall include the following components:

- Organization structure and responsibilities (included in Section 4.0-Responsibilities and included as part of the site-specific Health and Safety Plan-HASP),
- Comprehensive site work plan (contained in the site-specific HASP and site-specific Work Plan),
- Site specific HASP which includes an emergency response component (included in Section 5.2),
- Training program (included in Section 5.3),
- Medical surveillance program (included in Section 5.4), and
- Standard operating procedures (see Reference Section 6).

## 5.2 Health and Safety Plan (HASP) Development

HAZWOPER teams that work on a site-specific project must develop a HASP based on the site-specific work plan and address physical, chemical and biological hazards associated with the proposed work activities. The plan shall address the necessary engineering controls, administrative controls, and personal protective equipment required to mitigate the site hazards. For all ARCADIS HAWOPER work and unless otherwise required by clients, the E-HASP template is used as the basis of the site-specific HASP.

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In general, the HASP is required to contain:

- 1. A description of the work location, the site history, and a summary of any information available concerning site hazards (including both physical hazards and contamination conditions).
- 2. A summary of the work activities to be performed under ARCADIS' scope of activities.
- 3. A safety and health risk or hazard analysis for each on-site task, which will be performed. Identified risks must include both chemical and physical hazards to which personnel may be exposed during the conduct of the work task. (*NOTE: this is accomplished through the HARC process-see summary of the process below*).
- 4. Summarized protective measures for each work task to prevent or mitigate the potential hazards identified in the hazard analyses.
- 5. Summarized personal protective equipment (PPE) requirements.
- 6. Frequency and types of air monitoring, personal monitoring, and environmental sampling techniques and instrumentation to be used.
- 7. Site control measures.
- 8. Decontamination procedures (or references).
- 9. An emergency response plan addressing actions to be taken in the event of a credible incident which might result during the performance of planned work activities, including minor and major injuries, and chemical release and fire. Response plans must address the means for coordinating the evacuation of all on-site personnel in the event of a catastrophic incident.

It is preferred that there only is one HASP per work site. If multiple HASPs exist for a work site, then a copy of other contractor/subcontractor HASPs should be obtained by ARCADIS prior to starting work. Responsibilities of the client and site safety supervisors between HASPs should be coordinated and understood. In the event that there are conflicts between HASPs, the most conservative HASP should be followed.

The SSO is given authority to correct health and safety deficiencies of any contractor and stop work as necessary until deficiencies are corrected.

The HASP should include a signature page that contains signatures of those who wrote or edited the plan and, when required by a client or regulatory body, the signature of an authorized ARCADIS HASP Reviewer.

5.2.1 Hazard Assessment and Risk Control (HARC) and Monitoring

The HARC process is a tool to help evaluate the relative risks of tasks to determine the type of control to implement to minimize exposure. The HARC tool assists in assessing the hazards identified during the TRACK process, Job Loss Analysis (JLA) development, Tailgate safety meetings, PM/TM hazard analysis and H&S Planning, ranking hazards as high, medium, and low with standardized criteria. The process

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can be used to easily assess routine and non-routine hazards in the office and field environments. A full explanation on how to use the HARC process can be found in the ARCADIS H&S procedure – ARC HSMS002.

This process helps evaluate to determine ultimate control of hazards. In general, the following hierarchy of controls must be considered:

- Engineering Controls design and use of controls through physical means or modification to the work. Engineering controls also includes Substitution or Elimination – replacing or elimination of the hazard/exposure.
- Administrative Controls design and use of procedural steps, training, warning labels and employee rotation. For the purpose of the HASP examples include JLAs, using TRACK and providing appropriate orientation and training to employees.
- Personal Protective Equipment specification for and use of appropriate PPE to reduce and control exposures. Examples of PPE on HAZWOPER sites include respiratory protection for inhalation exposure, coated coveralls and chemical resistant gloves for dermal exposure and use of cooling vests for heat stress. Until such time that ARCADIS institutes the above controls or is such controls are not practical, feasible or effective, employees or subcontractors who are exposed to agents that have a potential for negative health effects and/or that are over occupational exposure guidelines (OEGs) will be required to wear appropriate personal protective equipment (PPE).

## <u>Monitoring</u>

The HASP must include a section on monitoring for potential hazards. This section shall include the frequency and type of monitoring to be performed; sampling techniques; maintenance and calibration of equipment; and information on instrumentation. Air monitoring may need to be conducted at each site prior to and during each work task. The results are used to determine actual employee exposures, the adequacy of designated protection levels, engineering controls and safe work practices. In addition, action levels must be established for each type of measurement taken. These action levels shall be based on the specifics of the work task and published exposure standards (PELs, TLVs, etc.). The most conservative value should be used if multiple values exist.

#### 5.2.2 Site Control/Decontamination Measures

Site control measures shall be developed in the work planning stage and modified as work progresses. Site control elements may include the following, but need not be repeated if covered under other parts of the HASP:

- A site map;
- Work zones and exclusion zones (See Exhibit 2) for information on these zones);

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- A buddy system;
- Site communications including a means to alert employees for emergencies; and
- Identification of the nearest medical assistance facility.

## Decontamination Procedures

Decontamination procedures shall be developed and implemented before employees or equipment enters areas of the work site where the potential for exposure exists. Procedures used are dependent on the level of contamination and the level of protection used by employees at the site. Decontamination situations will vary depending on the complexity of the site and chemicals of concern (COCs). Decontamination procedures shall be monitored by the SSO who will correct any deficiencies noted.

5.2.3 Emergency Response Plan

Regulations require that an emergency response plan be developed for work at all HAZWOPER, CERCLA (Superfund) and RCRA Corrective Action sites. As the emergency plan will be dependent on the site, COCs present, surrounding facilities, etc., the HASP must contain specific information addressing emergencies and equipment. In general, the plan shall include the following information not already contained elsewhere in the HASP:

- Methods and procedures for alerting employees to the emergency;
- Evacuation routed and procedures;
- Safe distances and places of refuge;
- Site security and control;
- PPE and emergency equipment.
- Additional elements may include general site topography and layout, prevailing wind direction, and procedures for reporting incidents to authorities. The plan requirements should be rehearsed regularly, reviewed periodically and amended as necessary to keep them current with site conditions and information. The plan shall be available for inspection and copying by employees and government.

## 5.3 Medical Surveillance Requirements

The medical surveillance program shall be incorporated into the HASP. Details of the medical surveillance program are included in ARC HSGE010.

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#### 6. TRAINING

Work shall not be performed by employees on HAZWOPER sites or on emergency response projects until they have been trained to the applicable level of their job function/responsibility. In general, the training will occur upon initial assignment and annually thereafter, and will cover:

- Recognizing hazards and how to prevent them;
- The names of personal and alternates responsible for site health and safety;
- Selection, care of and use respirators and other PPE;
- Engineering controls, medical surveillance requirements, emergency response procedures, spill containment procedures, confined space entry procedures, and any other work practices appropriate to a site; and
- Proper decontamination procedures.

See below for HAZWOPER and Emergency Response training requirements.

Staff	Hours of Training
Routine Site Employees	40 hours initial
	24 hours on the job
	8 hours annual refresher
Routine Site Employees	24 hours initial
(minimal exposure)	8 hours on the job
	8 hours annual refresher
Non-routine site employees	24 hours initial
	8 hours on the job
	8 hours annual refresher

#### Hazardous Waste Clean-Up Sites

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Supervisor/Managers o	<u>f</u>	Hours of Trai	ning
Routine site employees		40 hours initia	I
		24 hours on th	ie job
		8 hours Super	visors
		8 hours annua	l refresher
Routine site employees		24 hours initia	l
(minimal exposure)		8 hours field	
, , , , , , , , , , , , , , , , , , ,		8 hours Super	visor
		8 hours annua	l refresher
Non-routine site employe	es	24 hours initial	I
		8 hours field	
		8 hours Super	visors
		8 hours annua	l refresher
Treatment, Storage, and	d Disposal Sites		
<u>Staff</u>		Hours of T	raining
General Site Employees		24 hours initia	l or equivalent
		8 hours annua	l refresher
Emergency Response			
<u>Staff</u>		Hours of T	raining
Level 1 - First Responder	(awareness level)	Sufficient training or pr	oven experience in

Level 2 - First Responder (operations level)

Sufficient training or proven experience in specific competencies. Annual refresher *Note (1)* 

Level 1 competency and 8 hours initial or proven experience in specific competencies. Annual refresher *Note (2)* 

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Level 3 - HAZMAT Technician		24 hours of Level 2 and in specific competencie Annual refresher <i>Note</i>	s.
Level 4 - HAZMAT Specialist		24 hours of Level 3 and specific competencies Annual refresher Note (	
Level 5 - On-the-scene incident commander competencies		4 hours of Level 1 and Annual refresher Note	

#### Notes:

- 1. Witness or discovers a release of hazardous materials and who are trained to notify the proper authorities.
- 2. Responds to releases of hazardous substances in a defensive manner, without trying to stop the releases.
- 3. Responds aggressively to stop the release of hazardous substances.
- 4. Responds with and in support to HAZMAT technicians, but who have specific knowledge of various hazardous substances.
- 5. Assumes control of the incident scene is beyond the first-responder awareness level.

If an employee misses their anniversary date on their 8 hour refresher course, and that time is less than two years since they last had their refresher training, they can be certified by completing the 8 hour refresher course designated by Corporate H&S. If an employee has missed their refresher training for more than 2 years, the following criteria and actions apply:

- Greater than 2 years and less than 4 years The employee will complete the Competency Exam and if 90% is achieved, can take just the refresher; less than 90%, retake 40 hour training at a course designated by Corporate H&S
- Greater than 4 years: Retake the 40 hour course at a course designated by Corporate H&S

## 6. REFERENCES

ARCADIS Employee Field Health and Safety Handbook

ARC HSMS001 H&S Organization, Roles and Responsibilities

ARC HSMS002 Hazard Assessment and Risk Control

ARC HSGE010 Medical Surveillance

ARC HSGE015 Personal Protective Equipment

ARC HSGE017 Respiratory Protection

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# 7. Records

None

# 8. APPROVALS AND HISTORY OF CHANGE

Michael a Phomas

# History of Change

Revision Date	Revision Number	Reason for change
7 September 2009	01	Creation of Document

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Checked by:

1.

2.

3.

**Replacement:** Place an asterisk (\*) beside the date a missing item(s) was noted and when the vendor was called; note below when replacement was delivered. Include any other pertinent comments.

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# 1. POLICY

It is the practice of ARCADIS and its affiliated companies to implement appropriate, reasonable and practical procedures within acceptable and customary industry practices to promote the health and safety of its employees, and avoid and mitigate exposure of risk in the performance of their work. In furtherance of this policy, ARCADIS promotes and encourages compliance by all employees with this policy and procedures relating to subsurface work and/or investigations (SWI) and working in the vicinity of above ground utilities.

- This procedure is followed by all responsible ARCADIS personnel. Such procedures are included in the project planning processes utilized by ARCADIS personnel.
- Project management procedural requirements are outlined in Section 5.2. All employees included in SWI and above ground utility work are familiar with these procedures.
- Contract Terms: In agreements for SWI with a client, prime contractor, or subcontractors, required terms (Exhibit 1) shall be included for the appropriate allocation of risk of damage to subsurface facilities. If such provisions cannot be agreed upon, the reasons are documented and other risk-management actions identified, such as limits of liability, additional physical investigations, additional lines of evidence of utility location, assignment of risk to subcontractors, etc.
- The policy of ARCADIS encourages and empowers all employees to take such action as they deem appropriate to assure compliance with this policy and procedures both in project planning and field site operations. Such authority is delegated to those on the project site to immediately stop any SWI work or work in the vicinity of above ground utilities where the employee believes that injury to persons or damage to property could occur. Such action is taken without regard to costs or schedule. Personnel should immediately notify their supervisor of any concerns they have when observing any SWI work or work in the vicinity of above ground utilities. In all agreements between ARCADIS and SWI subcontractors, (e.g., drilling subcontractors), provisions shall be included in the subcontract, work authorization or purchase order. These provisions (Exhibit 1) are found on the ARCADIS intranet at the Legal Department team site.

All ARCADIS personnel involved in SWI work or work in the vicinity of above ground utilities will be appropriately trained on this procedure and have the appropriate professional experience for oversight of or involvement in SWI work or work in the vicinity of above ground utilities. ARCADIS Corporate Health & Safety can answer further questions about this policy or the hazards associated with and the control procedure for work in the vicinity of subsurface or above ground utilities.

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Again, to support the efforts of ensuring the health and safety of its employees and mitigating risk to ARCADIS, ARCADIS requires that these policies and procedures be followed and implemented at all levels of project management and field implementation.

# 2. PURPOSE AND SCOPE

#### 2.1 Purpose

This procedure directs general safety procedures associated with the identification and management of above ground and subsurface utility locations on project sites.

# 2.2 Scope

- 2.2.1 **Management Requirements** ARCADIS personnel managing or working on any project requiring SWI and requiring work in the vicinity of above ground utilities must incorporate this procedure into their project planning and field work activities to ensure that all reasonable means to identify utilities are implemented and that appropriate controls have been put in place to minimize or eliminate damage to these utilities and the hazards associated with these utilities. All applicable procedures described in this document must be completed prior to initiating intrusive field work or field work in the vicinity of above ground utilities, or the work cannot proceed.
- 2.2.2 **Project Management Requirements** Where SWI are required to be performed by a subcontractor to ARCADIS under its subcontract, project management shall require the subcontractor to adequately incorporate SWI procedures described herein into the subcontractor's scope of work.

#### 3. **DEFINITIONS**

**Above Ground Utilities -** For the purpose of this procedure, above ground utilities include, but are not limited to: any above ground line, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications, electricity, gas, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, or other hazardous materials.

**Subsurface Utilities -** For the purposes of this procedure, subsurface utilities include, but are not limited to: any underground line, system, or facility used for producing, storing, conveying, transmitting or distributing communication or telecommunications, electricity, gas, petroleum and petroleum products, coal slurry, hazardous liquids or gases, water under pressure, steam, or sanitary sewage; underground storage tanks; tunnels and cisterns; and septic tanks.

#### 4. **RESPONSIBILITIES**

#### 4.1 Project Manager Responsibilities

To prevent injury to employees, avoid disruption to utility services, and help eliminate damage to subsurface and above ground utilities, project managers have the responsibility for utility identification, location, and marking prior to initiating field activities. Most states, provinces, municipalities, and clients have rules, general statutes, or laws that specify the requirements of subsurface utility location prior to intrusive subsurface field activities (i.e., excavation, trenching,

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boring, and all forms of drilling operations, etc.). The project manager ensures that these laws are followed, and that the directives outlined in this procedure are met for every project involving SWI and work in the vicinity of above ground utilities.

In addition, if field activities are completed in the vicinity of above ground utilities, the project manager is responsible for working with the client to identify the nature of the utilities, and to determine what control processes need to be implemented to prevent damage to these utilities and to minimize any injury in the event there is damage.

#### 4.2 Field Personnel Responsibilities

Field personnel conducting SWI activities and activities where above ground utilities are in the vicinity of the work have the responsibility to read, understand, and follow this procedure and complete the appropriate checklists during the on-site utility locate process. ARCADIS personnel assisting in the identification of underground utilities need to have previous related experience of a minimum of 1 year. Those implementing remote sensing technologies must complete training in those techniques and have 6 months experience operating and interpreting results.

If utilities cannot be located to eliminate any reasonable concern, field personnel can use their Stop Work authority until utility locations can be identified. Field personnel must review this procedure onsite with ARCADIS subcontractors, and ensure they follow the procedures detailed in this document. Any ARCADIS subcontractor not following these procedures will be asked to stop work, and the project manager contacted. Any diversion from this procedure by ARCADIS field personnel must be approved by the project manager with input from Corporate Health & Safety as necessary.

#### 5. PROCEDURE

A flow chart/decision tree of the procedure is presented in Exhibit 2 of this document.

#### 5.1 Lines of Evidence

The following three actions (lines of evidence) are required for the utility location process:

- Contact the State One Call
- Obtain a detailed site utility plan drawn to scale, preferably an "as-built" plan
- Conduct a detailed visual site inspection

In the event that one or more of the above lines of evidence cannot be conducted, or if the accuracy of utility location is questionable, a minimum of one additional line of evidence must be utilized as appropriate or suitable to the conditions. Examples of additional lines of evidence include but are not limited to:

- Private utility locating service
- Research of state, county or municipal utility records and maps including computerdrawn maps or geographical information systems (GIS)
- Contact with the utility provider to obtain their utility location records

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- Hand augering or digging
- Hydro-knife
- Air-knife
- Radio Frequency Detector (RFD)
- Ground Penetrating Radar (GPR)
- Any other method that may give ample evidence of the presence or location of subgrade utilities

#### 5.2 Project Management Procedural Requirements

Field activities are planned and designed to avoid contact with and damage to, and minimize interference with subsurface and above ground utilities in the vicinity of ARCADIS work activities. During the planning phase of a project the project manager will insure the appropriate allocation of utility location responsibilities and verify their completion. The utility location activities will implement the lines of evidence as defined in Section 5.1.

#### 5.2.1 Communication and Coordination

The PM or their designated Task Manager:

- Communicates verbally and in writing the responsibilities for utility location with each party
- Provides the list to the site safety officer for inclusion in the site-specific health and safety plan (HASP);
- Communicates potential hazards to field staff prior to mobilization;
- Instructs field staff to be aware of and implement the procedures in the Section 5.1 of this procedure and utilize the appropriate utility location checklists.
- When practical, schedules a joint meeting between the public/private utility locators and field staff to oversee the subsurface utility locating and marking in the field.
- Communicates with and provides utility location documentation to the subcontractors to verify with them the utility locations and discusses methods to be used to protect those utilities.
- Understands the subcontractor's methods for utility location and documenting the process with a clear delineation of responsibilities for utility location.

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In general, subsurface utility locations marked by public utility locators are only good for 2 weeks (research your state-specific requirements). If SWI activities are not conducted during this time period, the site is remarked.

NOTE: At no time is SWI conducted based on old markings, hand-drawn maps/sketches, photographs, or by recollection/memory of field staff. If markings are smeared, removed, damaged, or impacted in any way, the site must be remarked before SWI begins. Flag markings are used in addition to paint markings wherever possible.

5.2.2 Utility Request Notifications for Public Property

Prior to intrusive work on public property (i.e., right-of-ways, easements, etc.), notification of a public one-call service center is completed a minimum of 48-72 hours (states/localities requirements vary, so the PM is responsible for verifying this) prior to initiating field activities (excluding Saturdays, Sundays, and legal holidays). Specific state or local laws related to utility location are evaluated with respect to notification and liability in the event of utility damage. During the call, the responsible party:

- Provides accurate description of the location of all areas of the SWI;
- Documents the utility locate request to record the time and date of the call, the area to be marked, the list of utility companies and municipalities that the one call service center will notify;
- Records the associated ticket (or dig) number provided by the one call service center;
- Cross references the notification list provided by the one-call service center with the list of known or suspected utilities for the property; and
- Provides accurate contact (responsible party name and phone numbers) information for the one call service center so they can subsequently communicate potential questions and/or delays related to the utility location and marking.

After receiving a request, the one-call service center sends requests to participating utility operators who have utilities in the area of the intrusive field activities. Each underground utility operator dispatches their own locators to mark their facilities with paint or flags. The project manager attempts to have field staff present during the marking of the utilities by the locator organization to ensure that the area of the SWI is included in the locating activities. It is important to note:

• Not all utility operators and municipalities participate in one call programs. In some instances, one-call programs provide a list of utility providers that participate, and a list of those that do not. The utility providers that do not participate are contacted individually so that they can

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mark their own lines, and this call is documented (date of call, person receiving call, date lines will be marked, etc.);

- Public utility locators are usually only required to mark utilities within the public spaces (i.e., right of ways) or at most up to a meter on private property; and
- Knowledge of existing or suspected, but unmarked utilities are documented and communicated to the site safety officer, field staff, and the client prior to implementing field activities.

If a known or suspected subsurface utility does not participate in the state one-call program, and that provider has not been individually contacted prior to the start of SWI, then the field activities are postponed. If these utility providers are contacted and do not provide utility location services, then SWI are not performed until a private utility locating company is contracted and the locating tasks completed.

#### 5.2.3 Nation-wi de Utility Locate Call Number 811

State and local utility notification centers participate in a "Call before you Dig" number for public safety and to protect underground infrastructure. This national number is: **811**. The number is designed to help prevent professional excavators, drillers and homeowners from damaging underground utility lines, or causing an injury or service outage while digging/drilling. For more information about the 811 services, visit www.call811.com

The number 811 is an FCC designated national n-11 number. This quick and efficient one call service will notify the appropriate utilities, who participate in the one call program. **However**, callers must still verify who the one call <u>service contacts</u>, and then determine which utilities may need to be contacted directly (e.g. those utilities not participating in the one call service) by following the requirements outlined in this procedure.

#### 5.3 Field Protocol

At no time do field activities that involve SWI or work in the vicinity of above ground utilities commence without the field staff having knowledge of the location of subsurface and above ground utilities. In addition, as stated above and in general, subsurface utility locations marked by public utility locators are only good for 2 weeks (research your state-specific requirements). If SWI activities are not conducted during this time period, all lines of evidence must be re-verified.

NOTE: At no time is SWI conducted based on old markings, hand-drawn maps/sketches, photographs, or by recollection/memory of field staff. If markings are smeared, removed, damaged, or impacted in any way, the site must be remarked before SWI begins. Flag markings are used in addition to paint markings wherever possible.

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#### 5.3.1 SWI and Subsurface Utilities

Prior to the start of intrusive activities, all utilities are located and measures instituted to avoid subsurface utility hazards. No SWI will be conducted within 30 inches of a line marking. If SWI must take place within 30 inches of the line marking, an additional line of evidence must be used that will ensure the avoidance of the line. An additional safety measure can include the use of lockout/tagout to render the utility controlled.

Prior to mobilizing to the site for SWI work, field staff reviews the task details with the project manager or their designated authorized TM. This may include but is not limited to review of boring logs, excavation permits, etc. Any special site or client requirements are also discussed. Prior to initiation of any intrusive activities, the utilities and structures checklist (Exhibit 3) is reviewed and completed. Generally, the following colors apply for different types of utilities/operations:

Red – Electric; Yellow – Natural gas/oil; Orange – Communication/cable television; Blue – Water; Green – Sewer; Pink – Temporary survey marking; White – Proposed excavation; and Purple – Reclaimed water

In addition, the SWI subcontractor marks (i.e., paint, stakes, etc.) the location of their operations to ensure they fall within the area that has been investigated for utilities.

Once the checklist is completed and all utilities identified, any client/site specific utility location or other utility (subsurface or above ground utilities) protection procedures (i.e. such as hand digging to a specified depth, covering or shielding lines, etc.) is completed at each location where work will be completed. If a known or suspected public subsurface utility has not been marked or the markings are not clear, the state one-call number is contacted to determine if an "emergency" locate can be requested. If so, follow the procedures outlined by the locate service and contact the project manager. If it is a private utility that is not marked, the facility manager and/or the project manager should be contacted.

If unexpected conditions are encountered (refusal, debris, pea gravel, etc.) while completing the intrusive activity, all work is immediately halted. Note that subsurface utilities at many industrial facilities are often placed in conduits or concrete to prevent damage. If a utility or subsurface structure is compromised, the field staff initiates the Emergency Action Plan Guidelines (Exhibit 5); however, more detailed emergency action procedures should be reviewed with the client and documented in the site specific health and safety plan prior to initiating work.

#### 5.3.2 Work in the Vicinity of Above Ground Utilities

If activities take place in the vicinity of an above ground utility, the utility line can be rendered controlled (i.e. through lockout/tagout procedures) or protected from damage (i.e. covering overhead power lines). The following table is used to develop acceptable work distances for work involving machinery with high extensions (backhoes, drilling rig masts, etc.) in the vicinity of overhead power lines:

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Power Line Voltage Phase to phase (kV)	Minimum Safe Clearance (feet)
50 or below	10
Above 50 to 200	15
Above 200 to 350	20
Above 350 to 500	25
Above 500 to 750	35
Above 750 to 1,000	45

ANSI Standard B30.5-1994, 5-3.4.5

The distance may be lengthened if directed by the client or the electric company, and any specified distances are strictly followed. In addition, work involving machinery, vehicles or equipment that may come in contact with above ground utilities is not completed until those utilities are protected or control processes are in place to avoid damage to those utilities.

If an above ground utility is discovered that has not been previously identified prior to mobilizing to the field, the field staff notifies the project manager who requests the client to assist in the identification of the utility and the implementation of control procedures as appropriate. In addition, if a utility or subsurface structure is compromised, the field staff initiates the Emergency Action Plan Guidelines (Exhibit 5); however, more detailed emergency action procedures should be reviewed with the client and documented in the site specific health and safety plan prior to initiating work.

#### 6. RECORDS

#### 6.1 Utilities Location Records

All records (maps and documentation of communications) used to determine the location of utilities should be retained and kept in the project file.

#### 7. APPROVALS AND HISTORY OF CHANGE

Approved By: Mija Coppola, Director H&S, Infrastructure and PM/CM Divisions

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# History of Change

Revision Date	Revision Number	Reason for change
13 December 2006	01	Original document
26 March 2007	02	Put in new company format
15 May 2007	03	Added nation-wide 811 number
6 September 2007	04	Changing over to new template format
22 February 2008	05	Changing over to new template format
13 January 2009	06	Define lines of evidence

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#### Exhibit 1 - Contract Term Language

# INSERT INTO ALL CLIENT CONTRACTS OR WORK ORDERS WHERE DRILLING, EXCAVATION, INTRUSIVE WORK IS TO BE PERFORMED.

**Site Conditions:** ARCADIS shall not be liable for: (i) damage or injury to any subterranean structures (including, but not limited to, utilities, mains, pipes, tanks, and telephone cables) or any existing subterranean conditions; or the consequences of such damage or injury, if (with respect to this clause (i)) such structures or conditions were unknown and were not identified or shown, or were incorrectly shown, in information or on plans furnished to or obtained by ARCADIS in connection with the Services; (ii) concealed conditions encountered in the performance of the Services; (iii) concealed or unknown conditions in an existing structure at variance with the conditions indicated by the Scope of Services or Work Authorization; or (iv) unknown physical conditions below the surface of the ground that differ materially from those ordinarily encountered and are generally recognized as inherent in work of the character provided under this Agreement.

Client shall provide to ARCADIS all plans, maps, drawing and other documents identifying the location of any subterranean structures on the Site. Prior to location of any drilling or excavation below the ground surface, ARCADIS shall obtain the concurrence of the Client as to the location for such drilling or excavation.

Should: (i) concealed conditions be encountered in the performance of the Services; (ii) concealed or unknown conditions in an existing structure be at variance with the conditions indicated by the Scope of Services or Work Authorization; or (iii) unknown physical conditions below the surface of the ground differ materially from those ordinarily encountered and generally recognized as inherent in work of the character provided under this Agreement; then the amount of this Agreement and/or time for performance shall be equitably adjusted by change order upon timely notice.

#### INSERT INTO ALL DRILLING, EXCAVATION, INTRUSIVE WORK SUBCONTRACTS.

**Site Conditions:** SUBCONTRACTOR acknowledges that time is of the essence with respect to the performance and completion of its work under this Contract. SUBCONTRACTOR shall adhere to, commence and complete its work in accordance with any schedule incorporated into this Contract, or any schedule submitted by SUBCONTRACTOR or attached hereto; and with respect to any Changes, out of scope or additional work, SUBCONTRACTOR shall expeditiously perform such work according to any schedule therefore agreed to by the parties. In the event any schedule is incorporated in this Contract or attached to this Contract, SUBCONTRACTOR acknowledges and agrees that such schedule has accounted for all inherent or reasonably anticipated delays, including but not limited to those inherent in obtaining site information, access sufficient labor, supplies, tools, equipment and utilities required for the project work, and SUBCONTRACTOR waives any claim of extra compensation or damages therefore.

Subcontractor represents and warrants that it has had an opportunity to review and/or has carefully examined all necessary drawings, maps, schematics, specifications, governmental restrictions, permits and license requirements, and all applicable laws, regulations and rules relating to the Work to be done and the Site, it surroundings and local conditions, and has made all investigations based on reasonably available information that are necessary to develop a full understanding of the hazards and difficulties which can be encountered and are likely to impact the cost or schedule to perform the Work. SUBCONTRACTOR is thus familiar with conditions at the Site as are pertinent to or which may affect the Work and has been granted the right to

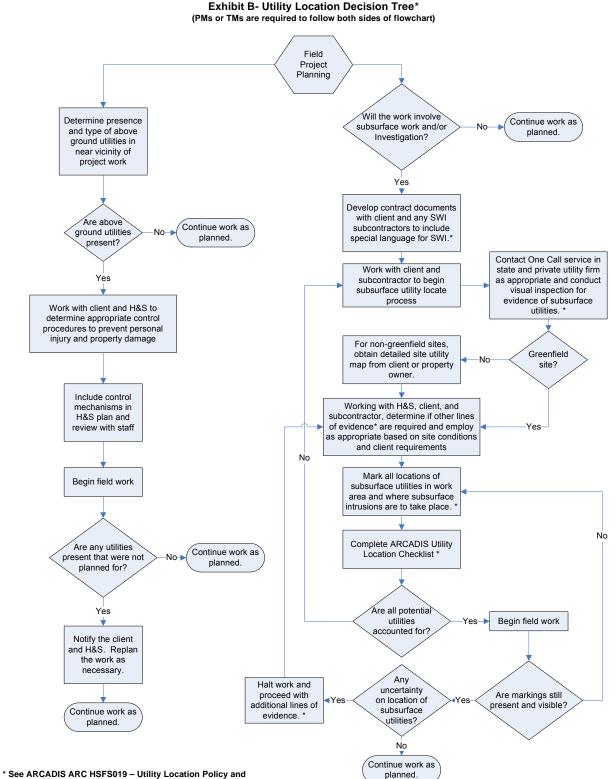
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conduct, and has conducted, all investigations it deems appropriate to determine that it can fulfill the requirements of this Contract. Notwithstanding any other provision of this Contract, SUBCONTRACTOR assumes the risk of all conditions, as specified in this Contract, that may affect SUBCONTRACTOR'S ability to perform the Work and will, regardless of such conditions, or the expense or difficulty of performing the Work or the negligence, if any, of ARCADIS, with respect to same, fully complete the Work for the stated price without further recourse to ARCADIS. Information on the Site and local conditions at the Site furnished by ARCADIS are not guaranteed by ARCADIS to be accurate, and is furnished only for the convenience of SUBCONTRACTOR.

The discovery of concealed conditions which could not reasonably have been anticipated by the SUBCONTRACTOR from information available to SUBCONTRACTOR may constitute a changed condition, which, to the extent such condition materially affects the cost or schedule to perform the Work, would entitle the SUBCONTRACTOR to a change and an equitable adjustment of the Contract price or time. SUBCONTRACTOR warrants that it shall conduct appropriate investigations to determine, with reasonable certainty, the location of utility and service lines, underground storage systems, and other subsurface structures of any kind before commencement of any drilling, excavation, or other work that has the potential to disturb these structures. SUBCONTRACTOR further warrants that it shall conduct independent field investigations to confirm the location of subsurface structures before commencement of subsurface work and shall not relay exclusively on plot plans or other drawings provided to SUBCONTRACTOR in conducting these investigations.

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#### Exhibit 2 – Utility Location Decision Tree



Procedure for full details.

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## Exhibit 3 - Utilities and Structures Checklist

Project:	Project Number:	
Site Location:	Date:	

**Instructions:** This checklist will be used as a safety measure to insure that all underground utility lines, other underground structures as well as above ground utilities are clearly marked out and identified in the area selected for boring or excavation. DRILLING, EXCAVATION, OR ANY TYPE OF GROUND INTRUSIVE WORK MAY NOT PROCEED UNTIL LINES ARE MARKED AND THIS CHECKLIST HAS BEEN COMPLETED.

Pre-Field Work Requirements		
Was the state one-call notified with the required advanced notice (usually 48 to 72 hours) (or 811 Nation-wide number)	YES NO	
State one-call confirmation number		
List utility companies who do not participate in the state one call program. Were they contacted directly?		
What additional lines of evidence are used for utility clearance?		
Was a plot plan showing site features and subsurface utilities provided by the PM/TM?	YES NO	
Subgrade Utility Line Location		
Where is the gas line located?		
Where is the gas meter located on the site building(s)?		
Are the electric lines subsurface or overhead? Where are they located?		
Where is electric meter located on the site building(s)?		

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Where are the telephone Are there any overhead		
Where do these lines en	ter the site building(s)?	
Where are the water line	s located?	
	se water (bathrooms, industrial uses, so where do the water lines enter the es?	
Are there small manhole so, where?	s/vault covers indicating water lines? If	
Was the local municipalit	y contacted to mark sanitary lines?	
Where are the sanitary li	nes located?	
	v lines enter the building? (i.e. what ne bathrooms, kitchens, water	
Where are the storm sev	ver lines located?	
Are there storm sewer in inlets for direction of sub	lets located on the property? Check surface lines.	
Are there any gutters direction of	ecting storm water to the subsurface? lines.	
Underground Storage	Fank Sites	
	ated? How many USTs are at the site counting fill ports and vent lines)?	
Where do the vent lines	run?	
Where does the piping ru to dispenser islands).	In? (Evaluate the path between USTs	
Where are the sub-surfa power to the UST system	ce electrical lines located which feed	
General Underground	Jtility Location Signs	
Are there any cracks res	embling straight lines that may indicate ?	

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Are there any patched a have been conducted?	areas where subsurface repairs may	
Are there any manhole associated with marked	covers or valve boxes that are not lines?	
Above ground Utility I	ine Location	
Are there overhead pov	ver lines? If, so where are they located?	
What is the voltage of the	ne overhead power lines?	
that are used by the clie the work area?	ound structures (utilities, piping, etc.) ent? If so, are they located proximal to	
Do these lines need con starting work?	ntrolled (locked out) or protected prior to	
	rs/Occupants MUST be interviewed utility lines at the site (if practicable)	
Name of Owner/Occupa	ant.	
How is this person affilia	ated with the Site?	
Who interviewed Owner	r/Occupant?	
Date of Interview		
Specific comments that	should be noted from the interview:	

NOTE: If any subsurface utilities listed above are not located, do not proceed with subsurface activities. Contact PM/TM immediately.

Name and signature of person who conducted utility line checklist

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#### Exhibit 4 - Use and Limitations of Utility Locating Methods

#### Ground Penetrating Radar (GPR)

The GPR system transmits high frequency electromagnetic waves into the ground and detects the energy reflected back to the surface. Energy is reflected along boundaries that possess different electrical properties. Reflections typically occur at lithologic contacts or where subsurface materials have high electrical contrasts, including metal objects such as underground storage tanks (USTs), drums, and utility pipes. These reflections are detected by the antenna and are processed into an electrical signal that can be used to image the subsurface feature. The GPR data will be reviewed in the field to assist in the delineation of potential piping or other subsurface structures.

The detection of subsurface structures located at the site depends on the electrical properties of the soil and the structure's depth, diameter, and composition. GPR is limited to the detection of smaller diameter pipes with depth. Generally, a pipe must increase in diameter by one 1 inch for each foot in depth to be seen using GPR. Also, plastic piping is more difficult to detect than metal piping using GPR, and caution should be used if plastic utility lines are suspected.

#### Radio Frequency Detection (RFD)

This instrument operates on the principle of radio frequency transmission and detection. The transmitter applies a known frequency to the pipe and the receiver is able to detect this frequency along the length of the structure. The success of RFD in tracing underground utilities is based on the composition of the structure (metal or plastic) and the ability to accurately position the transmitter unit so that it can be attached to, or placed directly over the structure. RFD should only be used to verify the location of utility mark-outs, and not as the primary method of utility identification.

#### **Soil Vacuum Excavation**

This method uses nondestructive vacuum excavation methods to create a visual test hole allowing the confirmation of buried utilities. This method is very accurate and relatively fast and can be performed prior to or during the drilling program. The limiting factors for this method are cost and availability. As with specialty drilling methods, a limited number of firms have the equipment to perform vacuum excavation.

The location of the structures to be cleared relative to the source and depth of impacted soil or groundwater is considered. If the zone to be cleared is known not to contain hazardous vapors or petroleum hydrocarbons via previous testing, continuous air monitoring is implemented using a lower explosive level (LEL)/O2 meter and photoionization detector (PID) or flame ionization detector (FID) to the depth of the boring. Also consistent with the site health and safety plan (HASP), air monitoring should be conducted continuously with the LEL/O2 meter during any activity if flammable or explosive vapors are suspected to be present. Prior to any subsurface investigation activities, air monitoring should be conducted to establish background levels for total organic vapors using a PID or FID. All work activity must STOP where tests indicate the concentration of flammable vapors exceeds 10% of the LEL, and the source of vapors must be investigated.

Vacuum-assisted soil excavation utility clearance will not be used in areas know to contain hazardous vapors or petroleum hydrocarbons unless the equipment to be used is suitable for flammable/explosive atmospheres. There is a significantly increased risk of explosion if these

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materials are encountered while performing this type of utility clearance. Cautions will be performed, as identified below.

#### Cautions

Many vacuum systems that are commonly used for utility clearance are considered unsuitable for use for environmental investigation sites. Most vacuum units are "Not for use with Hydrocarbons, Explosives, Corrosive or Toxic Material," and are "Not Intrinsically Safe."

Given that many units and associated tanking are not explosion-proof, the following steps will be considered prior to using vacuum- assisted utility clearance units where soils could be impacted with petroleum hydrocarbons or flammable vapors.

- 1. Request from the manufacturer and/or the contractor doing the work to supply manufacturers' documentation and specifications for use of the unit at environmental sites.
- 2. Request documentation that the unit is intrinsically safe and may be used in areas where petroleum hydrocarbon may be present.
- 3. Obtain the procedures for grounding portable units to discharge potential static electricity during operation.
- 4 If none of the above are available, then hand auger instead and do not use vacuumassisted methods.

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#### **Exhibit 5 - Emergency Action Plan Guidelines**

When work activities result in the contact or compromise of a utility line, an appropriate response is critical to prevent injury, death or significant property damage. Although circumstances and response vary depending on site specific conditions, the following guidelines provide information that is factored into emergency action planning associated with utility damage. In any event, emergency planning is coordinated with the entity that owns the utility and the client prior to the start of work. This planning and the appropriate response actions are documented in the project health and safety plan and reviewed with all field staff.

#### **Contact with Above or Underground Electric**

Contact with above ground or underground electric lines may result in the equipment being energized. Field personnel do not assume rubber tires on equipment are insulating the equipment from the ground. For underground electric strikes, contact with the line may not be immediately noticeable but indications of a strike include: power outage, smoke, explosion, popping noises, or arching electricity. If contact with an electric line is made or is suspected, the following guidelines are followed:

- Under most circumstances, the equipment operator or any worker on a seat of the equipment should stay on the equipment. These workers should not touch anything, especially metal, on the equipment.
- If it is determined that the equipment should be vacated due to a life threatening circumstance, the worker(s) should jump clear as far as possible from the equipment. When jumping keep both feet together and hop away to a safe distance after landing on the ground. Do not use hand holds or touch any part of the equipment when preparing to jump off.
- · Workers on the ground should move away from the equipment.
- Keep others away from the equipment and the area.
- If anyone is injured and in contact with the line or equipment, any attempted rescue should be performed with extreme caution. Only use long, dry, clean, unpainted pieces of wood or fiberglass pole or long dry, clean rope to retrieve the victim. Perform first aid/CPR only after the victim is sufficiently clear from the electrical hazard.
- Notify the electric utility or the client as appropriate for the site. Call 911or the client's emergency response phone number, as appropriate, for any serious injury or any situation that may result in fire or other hazard that could produce injury or property damage.

#### Natural Gas

If a natural gas line of any size is compromised, immediately:

- Shut off the equipment and remove any other ignition sources.
- Evacuate the area as quickly as possible.

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- DO NOT attempt to turn off any gas valves.
- Call 911 or the designated client emergency response number as appropriate.
- Call the gas utility, if site response is not controlled by the client.
- Do not return to the area until permitted by the utility or by the approved client emergency response personnel, as appropriate.

#### Water Lines (all types)

Compromised water lines may rapidly become a significant hazard especially if the line is under considerable pressure. Ruptured pressurized water lines may undermine and wash out unconsolidated materials beneath equipment or structures causing them to become unstable. If a pressurized water line is ruptured, the following guidelines should be followed:

- Promptly shut off all equipment.
- Lower masts or other high extension components of the equipment.
- Evacuate area and call the water utility or client emergency response number, as appropriate.
- Turn off the water if the valve location is known and on the site property.
- If potable water lines have been ruptured, attempt to divert any flow away from structures prone to being flooded. Use caution and keep a safe distance from the line break since the ground surface may be compromised.
- For raw process water or other water of unknown quality, do not attempt to divert or contain. Avoid skin contact or accidental ingestion of any water.
- When returning to the area of the break, survey the area for signs of compromised land surface (cracks in asphalt or concrete, depressions in ground, observations of undercutting, etc.) and avoid moving any equipment until these conditions are repaired or resolved.

#### Sewers (all types)

Use the same general guidelines for water lines when responding to compromised sewers. If a sanitary sewer is compromised additional guidelines should be followed to avoid contracting any bacterial illnesses. These include:

- Promptly evacuate the area.
- Avoid contact with any sewage material.
- If contaminated, promptly was with soap (antimicrobial) and water and promptly change impacted clothing.

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- If sewage is accidentally ingested or infiltrates any breach of the skin or enters the eyes, seek medical attention as a precautionary measure.
- Decontaminate equipment with commercially available disinfectant solutions or a 10% chlorine bleach solution.

#### **Communication Lines**

Contact and compromise of communication lines are generally considered more of a financial concern than a concern associated with injury. However, eye damage may occur if looking into the ends of a cut fiber optic line. Do not look into the ends of fiber optic lines or other communication lines of unknown type. Promptly contact the communication company owning the line.

#### Product Lines and Underground Storage Tanks (all types)

Compromise of a product line or underground storage tank (UST) requires immediate action to mitigate impact to the environment. For gasoline stations and similar facilities the following guidelines should be followed during a line or UST breach:

- Immediately shut down equipment and turn off the emergency shutoff switch for the facility dispensers.
- If there are no injuries, attempt to contain any flowing product using absorbent materials and/or by physically pumping or bailing product out of the breached area.
- If product is flowing on the surface away from the break area, attempt to protect downgradient storm drains, sewer drains, and surface water features form impact of the petroleum product using any readily available materials.
- If the bottom of a UST has been breached, immediately contact a pump truck to remove product from the affected UST.
- For releases involving diesel fuel, care will be taken to avoid any situation where diesel may be injected into the body from impalement by coated nails, wood splinters, etc. If diesel is injected into the body, seek prompt medical attention, even if no apparent symptoms of a problem exist.
- Clear area and arrange for prompt repair.

For industrial sites with lines or USTs containing multiple products with varying hazards, similar guidelines may be followed as above if the material encountered is known and workers have a fundamental understanding of the hazards associated with the material. Upon discovery of a line or UST breach due to work activities at these sites:

• Immediately stop work and notify the client representative or call the client designated emergency number. For abandoned sites call 911.

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• If the material is not known, promptly evacuate the area and let HAZMAT teams deal with the release.

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#### 1. Policy

ARCADIS understands the hazards of personal exposure to benzene. Based on this understanding, ARCADIS will implement the appropriate controls to minimize or eliminate the hazards of benzene. These controls will focus first on engineering controls to mitigate benzene hazards where appropriate and practical. Administrative controls may also be implemented as appropriate and practical. Where it is not appropriate or practical to implement engineering and administrative controls, personal protective equipment (PPE) will be implemented to control benzene hazards below known occupational exposure limits.

#### 2. Purpose and Scope

#### 2.1 Purpose

- 2.1.1 Benzene Exposure Protection This policy and associated procedures provides information to protect ARCADIS employees, subcontractors, and other effected personnel from exposures to benzene while conducting work on ARCADIS projects.
- 2.1.2 OSHA Requirements This policy meets the requirements of the U.S. Occupational Safety and Health Administration (OSHA) regulations including Title 29 Code of Federal Regulations (CFR) Part 1910.1028.

#### 2.2 Scope

This policy and the associated procedures apply to all projects where benzene is known or thought to be present, and where ARCADIS employees, subcontractors and other effected personnel are or could be exposed to benzene above the Action Level.

#### 3. Definitions

**Benzene**—is a colorless liquid with a sweet odor. It evaporates into the air very quickly and dissolves slightly in water. It is highly flammable and is formed from both natural processes and human activities. Some industries use benzene to make other chemicals which are used to make plastics, resins, and nylon and synthetic fibers. Benzene is also used to make some types of rubbers, lubricants, dyes, detergents, drugs, and pesticides. Benzene is also a natural part of crude oil, gasoline, and cigarette smoke.

Benzene is encountered on ARCADIS projects, frequently, as a contaminant in soils, ground and surface water, sediments, and other environmental media. Personnel may also encounter benzene in other forms at certain client facilities at which ARCADIS works. It can be encountered at petroleum-related facilities, chemical production facilities and other types of industrial sites.

Action Level—the airborne concentration established by OSHA that triggers certain regulatory requirements.

HSP—Health and Safety Procedure

**Permissible Exposure Limit (PEL)**—an average airborne concentration regulatory limit established by OSHA above which requires control to protect people from adverse health effects.

**Short Term Exposure Limit (STEL)**—a PEL or TLV established as a limit of exposure measured over a designated period of time less than 8 hours.

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**Threshold Limit Value (TLV)**—a recommended average airborne concentration limit established by ACGIH. The TLVs are reviewed and updated as appropriate annually.

**Time Weighted Average (TWA)**—a measurement of airborne exposure to a chemical compound measured and averaged over a designated period of time for comparison to an STEL or an 8-hour PEL or TLV.

#### 4. Responsibilities

- **4.1 Principal-In-Charge, Project and Task Managers** are responsible, as part of the project hazard assessment, for determining if benzene is or is potentially present on a project site. In addition, the project or task manager is responsible for determining client requirements with respect to the control of benzene hazards. Project and Task Managers notify health and safety staff when working on sites containing benzene. Project and Task Managers are also responsible for ensuring that project staff has the appropriate and applicable training for benzene prior to those staff beginning work.
- **4.2 Corporate Health and Safety** is responsible for keeping this policy and procedure up-todate with current regulatory requirements and best practices. In addition, Corporate Health and Safety oversees the medical surveillance program for benzene, as applicable and provides a benzene training package for presentation to appropriate staff.
- **4.3 Project Health and Safety Staff** including designated Writers and Reviewers of Project Health and Safety Plans (HASPs) are responsible for developing control processes and techniques on specific projects based on the levels of benzene expected to be encountered on project facilities.
- **4.4 Project Personnel** are responsible for completing benzene training as required by this policy and procedure, and for following all hazard control processes designated by the Project Manager, Project Health and Safety Staff, and the project HASP. If project personnel believe that benzene is present that was not previously identified or is at levels that are higher than expected, they should stop work and notify project health and safety staff or the project manager immediately and not proceed until authorized.

#### 5. Procedure

#### 5.1 Benzene Hazards

- Benzene is primarily an inhalation hazard. Benzene vapor does not present an appreciable skin hazard; benzene liquid is absorbed through the skin.
- The acute (short term) effects of inhalation exposure are similar to most other hydrocarbons (narcosis, dizziness, weakness, headache, nausea).
- Prolonged or repeated exposure to concentrations above the permissible exposure limits may lead to blood disorders, including anemia, leucopenia (low white blood cell counts), and leukemia (cancer of the blood system).

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 As with most hydrocarbons, repeated/prolonged skin exposure to liquid may lead to the aforementioned disease(s) of the blood.

## 5.2 Exposure Limits and Regulated Areas

The following personal exposure limits are established for benzene by inhalation:

• OSHA ACTION LEVEL – 0.5 ppm benzene in air 8-hour TWA.

# OSHA PELs

- TWA 1.0 part per million (ppm) benzene in air averaged over an 8 hour period.
- STEL 5.0 ppm benzene in air averaged over any 15 minute period.
- ACGIH TLVs
  - TWA 0.5 ppm benzene in air averaged over an 8 hour period
  - STEL 2.5 ppm benzene in air averaged over an 8 hour period
  - Skin notation meaning that there is a significant contribution to overall exposure by the cutaneous route including mucous membranes and the eyes, and by contact with vapors, liquids and solids containing benzene.
- Personal exposure is the concentration of benzene to which a person would be exposed if that person were not wearing respiratory protection. Personal exposures shall be measured over the exposure period in the breathing zone of the employee. Personal exposures should not be determined by area sampling.
- REGULATED AREA
  - An area where the benzene exposure does or can be expected to exceed the PELs or TLVs. Since it may be difficult to determine the exposure time for employees working in areas with concentrations that exceed PEL or TLV values, the facility/location may wish to regulate any area that exceeds 0.5 ppm or per the requirements of the client or of the project HASP.
  - The PEL for benzene is relatively low as compared to the PEL or TLV of other hydrocarbons such as gasoline (300 ppm); therefore, depending on exposure conditions, it may be very "easy" to exceed the PEL or TLV for benzene even though other hydrocarbon levels are not considered very high. Also of concern is historic monitoring data that indicates that short term work activities such as draining a cargo hose of gasoline or pumping free product from an aquifer may result in a benzene exposure exceeding the STEL.

# 5.3 Actions for Employee Exposures Greater Than or Equal to the OSHA Action Level or ACGIH TLV – TWA but Less than the OSHA PEL - TWA

• Training: Annual benzene training is required.

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- Respiratory Protection: full-face air purifying respirators equipped with organic vapor cartridges will be used per the project HASP.
- Medical Surveillance: Initial and annual medical exams (see below) are required if employee personal exposures do or can be reasonably expected to exceed the Action Level on at least 30 calendar days during the coming year.
- Periodic Monitoring shall be conducted at least annually until at least two consecutive exposure determinations (no less than 7 days apart) indicate the exposure is below the Action Level.

# 5.4 Actions for Employee Exposures Greater Than PELs

- Respiratory Protection: respirators shall be used in all regulated areas.
- Training: Annual benzene training is required.
- Medical Surveillance: Initial and annual medical exams (see below) are required if employee personal exposures do or can be reasonably expected to exceed the PEL on a least 10 calendar days during the coming year.
- Written Program: A written program to reduce personal exposure is required detailing the methods to be used to reduce exposures below the TLVs and the OSHA Action Level. These written programs will be in the form of the project HASP based on project-specific and client requirements. The HASP will indicate the schedule for the implementation of the any benzene-related hazard control processes or methods. The HASP is reviewed periodically per the ARCADIS HSP ARC HSFS010 – Health and Safety Plans. All project personnel have access to the project HASP at all times.
- Periodic Monitoring at least every 6 months until at least two consecutive exposure determinations (no less than 7 days apart) indicate the exposure is below the PEL; then annually until at least two consecutive exposure determinations (no less than 7 days apart) indicate the exposure is below the PEL Action Level.

# 5.5 Exposure Monitoring

- Representative personal exposure monitoring is required for each type of operation involving the handling of or potential exposure to benzene.
- Personal exposure monitoring shall utilize standard industrial hygiene sampling techniques and recordkeeping.

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- Passive badges such as the 3M 3500 or charcoal tube sampling may be used for this sampling activity.
- Detection tubes shall not be used for compliance personal exposure determination but may be used for work and confined space entry permitting and defining regulated areas.
- Employees who have been monitored for benzene exposure shall be notified of the monitoring results within 15 working days of receipt of these results. If the PEL is exceeded, the notification must indicate the follow-up plans or corrective actions to be taken to reduce exposures to below the PEL.
- Personal STEL monitoring should be used to characterize exposures for specific tasks such as gauging, O&M of treatment equipment, hose connect and disconnect, maintenance tasks such as flange breaking, etc.
- Personal TWA monitoring can be used for extended tasks, such as well developing and sampling, loading, tasks inside vessel holds, tank cleaning, and maintenance tasks such as pump removal, etc.
- Area sampling can be used to determine regulated areas; the sampling media shall determine the duration of sampling:
  - Detection tubes (Kitagawa #118SB, or Draeger 0.5/c) can be used for real-time determination.
  - Charcoal tube samples must be taken for at least 15 minutes (passive badges are not recommended for area sampling).
- Periodic Monitoring is required if exposures exceed the Action Level or PELs.

#### 5.6 Requirements for Regulated Areas

• Posting: Regulated areas shall be indicated such as by barricades, barricade tape, painted demarcations, or other devices.

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• A sign shall be posted at the access to the regulated area with the warning:

	1. DANGER
	2. BENZENE
	3. CANCER HAZARD
4.	FLAMMABLE - NO SMOKING
5.	AUTHORIZED PERSONNEL ONLY

[Minimum lettering height: DANGER BENZENE 4"; others 3"]

- Respiratory Protection: Respirators shall be worn by all personnel when in a regulated area, regardless of the time period or over-all personal exposure measurement.
- Labeling
  - In addition to appropriate Hazard Communication labeling, containers or equipment containing > 0.1% benzene must also be labeled as such:



– Pipelines do not need to be labeled.

#### 5.7 Exposure Reduction

- Written Program
  - The Project Manager and the Project Health and Safety Staff will develop a written program for exposure reduction if there is a determination that employee exposures may exceed the PELs or TLVs.
  - The written program must list the corrective actions that will be taken to reduce employee exposure to at or below the PELs and TLVs:
    - identify regulated areas/tasks;
    - engineering controls;
    - revised work practices;
    - respiratory protection and protective clothing; and
    - schedule of development and implementation.

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• Spills and Emergencies

An emergency is any occurrence which may result in an unexpected significant release of benzene that may result in a significant inhalation or skin exposure. After an emergency, appropriate monitoring must be conducted to assure the ambient benzene levels are back to normal; and conduct appropriate medical surveillance for affected employee(s).

- Respiratory Protection and Personal Protective Equipment
  - Respirators shall be worn, maintained and managed in accordance with the OSHA standard, 29 CFR 1910.134 and ARCADIS HSP ARC HSGE017 Respiratory Protection. In addition, any client requirements on project sites will be followed.
  - Per the project HASP, respiratory protection will be worn at all times when airborne concentrations of benzene exceed the OSHA Action Level or the ACGIH TLV-TWA. The respirator will be a full-face air purifying respirator equipped with organic vapor cartridges. Action limits for upgrading to a higher level of protection will be documented in the project HASP or per client requirements.
  - Appropriate eye protection will be worn as necessary. Protective clothing and gloves suitable for the particular product (such as for gasoline) will generally be suitable for protection against the benzene in that product. For most hydrocarbon products, nitrile gloves, provide adequate protection. Chemical resistant clothing may vary depending on the product and degree of exposure.
  - For "pure" benzene the following materials are recommended:
    - gloves: poly-vinyl alcohol (PVA)
    - clothing: Saranex or Barricade (DuPont) or equivalent.

# 5.8 Medical Surveillance

- Initial medical surveillance is required:
  - If employee personal exposures are reasonably expected to exceed the Action Level on at least 30 calendar days per year; or
  - If employee personal exposures are reasonably expected to exceed the PEL on a least 10 calendar days per year.
- Periodic exams are required on an annual basis for employees who continue to meet the criteria listed above. Annual exams may be discontinued after the exam conducted the year after personal exposures fall below the limits stated above in this section.
- The specific medical exam requirements are explained in detail in ARCADIS HSP ARC HSGE010 Medical Surveillance.
- The physician must be supplied a copy of the OSHA benzene regulation 29 CFR 1910.1028 and a description of the employee's benzene exposure.

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- For employees exposed to benzene from an emergency, a urine sample must be taken at the end of the shift. A urinary phenol test must be performed on the sample within 72 hours.
- OSHA regulations for benzene have specific medical removal provisions for medical examinations results falling outside of certain criteria. The facility/location should contact the Corporate Health and Safety Manager if the examining physician indicates that an employee may fall into these criteria.

# 5.9 Training

- Initial benzene training is required for all employees assigned to a work area suspected or known to contain benzene.
- Annual benzene training is required for all employees actually or potentially exposed to greater than the Action Level (TWA > 0.5 ppm).
- Initial and annual training shall consist of:
  - The operations that involve benzene exposure.
  - The methods/observations that can be used to detect the presence or release of benzene
  - The physical and health hazards of benzene.
  - Methods used to protect against the hazards of benzene.
  - The proper use of personal protective equipment in emergency situations.
  - The meaning of a regulated area and how such are demarcated.
  - A review of the applicable standard and where copies can be found.
  - An explanation of the medical surveillance program
- 6. References
- OSHA 29 CFR 1910.1128 Benzene
- ACGIH 2006 TLVs and BEIs Threshold Limit Values for Chemical Substances and Physical Agents & Biological Exposure Indices
- ARCADIS Medical Surveillance HSP ARC HSGE006

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## 7. Records

- All exposure, medical, and training records shall be kept for 30 years.
- All exposure and medical records shall be made available to appropriate regulatory agencies upon written request.
- Employees who have been monitored for benzene exposure shall be notified of the monitoring results within 15 working days of receipt of these results; a written request is not required

# 8. Approvals and History of Change

Approved By: Mija Coppola, Director H&S Compliance Assurance and LPS

Mija A. Coppola

#### **History of Change**

Revision Date	<b>Revision Number</b>	Reason for change
26 March 2007	01	Original document
7 June 2007	02	Change to new template
6 September 2007	03	Changing over to new template format
22 February 2008	04	Template change

Infrastructure, environment, facilities	ARCADIS HS Procedure Name Hearing Conservation Health & Safety Procedure	<u>Revision Number</u> 02
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## 1. POLICY

It is the policy of ARCADIS to assess noise hazards resulting from or encountered by our staff during job activities and to control such noise hazards to minimize and eliminate hearing loss among our staff, subcontractors, clients, and the public. Any employee who will be exposed to noise at or over 85 decibels (excluding brief intermittent ambient noise) for any amount of time will be required to wear appropriate hearing protection. When in doubt, ARCADIS will provide hearing protection.

# 2. PURPOSE AND SCOPE

#### 2.1 Purpose

ARCADIS is committed to providing a healthy and safe work environment for our employees, subcontractors, clients and visitors. To this end, ARCADIS embraces this Hearing Conservation Health & Safety (HS) Policy. The purpose of the ARCADIS Hearing Conservation HS Policy is to provide a standard policy on the health and safety requirements and processes for all employees with potential exposure to excessive noise (levels in excess of 85dBA for any amount of time) and to comply with 29CFR1910.95. ARCADIS defines excessive noise as any noise environment that requires speech levels above those used for normal conversation.

# 2.2 Scope

This policy and associated procedures apply to every project and all operations conducted by ARCADIS. Hearing Protection is supplied and/or approved by ARCADIS for use by employees in carrying out their assignments. All employees conducting work where the potential for excessive noise is present, are required to have their assigned hearing protection available and used as required by the project Health and Safety Plan (HASP), Job Safety Analysis (JSA), or client requirements.

#### 3. DEFINITIONS

**NRR** – Noise Reduction Rating is the measure, in decibels, of how well a hearing protector reduces noise, as specified by the Environmental Protection Agency. The higher the number, the greater the noise reduction. When dual protectors are used, the combined NRR provides approximately 5 decibels more than the higher rated of the two products. For example, using ear plugs (NRR of 29 decibels) with ear muffs (NRR 27) would provide a Noise Reduction Rating of 34 decibels. For practical purposes, users should assume they will actually receive protection that is 5 decibels less than the published value.

HSP - Health and Safety Procedure

**TWA** – Time Weighted Average; The average exposure to a contaminant or condition (such as noise) to which workers may be exposed without adverse effect over a period of 8 hours a day or a 40 hour work week.

**Decibels – A Weighted –** the unit of measure to be used when measuring noise levels on ARCADIS work sites and when comparing to occupational exposure standards and limits.

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#### 4. **RESPONSIBILITIES**

**Employees** – are required to wear prescribed hearing protection during activities with identified excessive noise levels. In addition, employees are required to have their provided hearing protection available where the potential for excessive noise exists and to use as required by HASPs, JSAs, or client requirements.

In addition, employees have the responsibilities to adhere to this HSP and to communicate HS concerns, issues and questions to their supervisor or their respective Health and Safety resource. In addition, all employees have the responsibility to:

- Use the TRACK process prior to any activity;
- Follow all ARCADIS and client requirements;
- Participate in the medical monitoring program, including annual audiogram and hearing conservation training as applicable based on their job duties;
- Notify the Corporate Health and Safety if they were exposed to high noise levels and required to wear hearing protection during the previous year and are not already in the medical monitoring program;
- To understand and appropriately utilize the "Stop Work Authority" concept.

**Managers** – Have the responsibility to steward the HS program to ensure that staff in their practice are appropriately equipped with the necessary hearing protection and have been provided the appropriate training. To accomplish this, Business Practice Managers (BPMs) have the responsibility to know and understand our HS program, policy, vision, and this HSP in detail enough so as to be prepared to explain it to a client when required. In addition, Managers have the responsibility to provide oversight management for the HS of employees in their respective operations. Each will assure that appropriate time and resources are provided to facilitate the implementation of this HSP. In addition, the Managers will involve themselves in any "Stop Work" issued by an employee as requested by an ARCADIS employee, project manager, or Principal-in-Charge (PIC). Managers will assist in resolving the issue associated with the "Stop Work Authority" issued by an employee.

**Project Managers and Principals in Charge (PICs)** – Have the responsibility to know and follow all applicable ARCADIS and client HS requirements, for ensuring work is conducted under the policy stated in this HSP, and for implementing the procedure requirements provided for in this HSP on any project and/or in offices that pose hazards to ARCADIS employees or employees of its subcontractors, clients, and other organizations present in the vicinity of work controlled by ARCADIS

For project related work, Project Managers and PICs responsibilities also include determining and communicating any specific client requirements that are applicable, including:

 Communicating with and appropriately managing subcontractors, ensuring that employees have appropriate training and qualifications, and for ensuring all client HS requirements are met;

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- Involving the appropriate ARCADIS HS Staff and project client staff, as necessary;
- Ensuring that all subcontractors have been communicated with concerning the minimum HS requirements for the project
- Providing adequate resources and budget for personal protective equipment (PPE), including hearing protection; PPE will be provided at not cost to the employee

In addition, as project and client agents and on behalf of ARCADIS, the Project Managers and Client Managers for client-related work shall be responsible for:

- Understanding and compliance by employees with HS rules and the requirements;
- Guaranteeing each employee the absolute right to exercise "Stop Work Authority" in good faith without fear of retribution or disciplinary action
- Using the ARCADIS Incident Investigation process for formally resolving a "Stop Work" condition.

Using this "Stop Work Authority" process, the manager and the employee will:

- Discuss and document the condition;
- Identify and document the root cause for the condition;
- Determine and document the solutions;
- Implement the solutions;
- Sign and acknowledge the solutions are in place to the satisfaction of the employee.

Corporate HS Staff – Have the responsibility for:

- Communicating the policy and procedure requirements in this HSP with all offices within ARCADIS – US;
- Ensuring that offices are aware of this HSP;
- Ensuring this HSP is being implemented effectively;
- Provide required training or guidance on approved training options;
- Providing the necessary suppliers and criteria for selection of H&S equipment.

**Health and Safety Managers and Specialists** – Are responsible for facilitating the policy and procedure requirements in this HSP in their area of responsibility and for providing "hands-on" assistance to ARCADIS staff to ensure this procedure is appropriately implemented.

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#### 5. PROCEDURE

#### 5.1 Noise Monitoring and Exposure Assessments

Noise monitoring should be conducted on any or all activities where excessiv e noise may be present. The monitoring will be prescribed by H&S professionals during the development of HASP and/or JSA. Noise monitoring may also be conducted at the discretion of the health and safety supervisor (HSS) or any staff members that hav e questions or concerns about potential noise exposure. ARCADIS defines excessiv e noise as any noise environment that requires speech levels above those used for normal conversation. If noise monitoring is not feasible, the staff will assume that exposures that require elevated speech are above 85 db and will use appropriate hearing protection. Monitoring results will be collected in accordance with guidance provided in 29CFR1910.95 Appendix G - Monitoring noise levels non-mandatory informational appendix. Monitoring results will be communicated to staff and used to determine adequate types and effectiveness (NRR) of hearing protection.

Community based noise monitoring may also be required based on the scope of the project. Community based noise monitoring will be conducted in accordance with the Project specifications and applicable Environmental Protection Agency (EPA), State or Local ordinances.

#### 5.2 Audiometric Testing

Audiometric tests will be scheduled in conjunction with pre-placement, periodic, and termination medical examinations as required by the Medical Surveillance Program. All employees that are not already part of the medical monitoring program must inform their supervisor and Corporate Health and Safety if they were exposed to high noise levels as part of their job duties. Employees that were exposed to high noise levels must receive an audiogram as specified by the Medical Surveillance Program. Employees will be informed of the requirement that they avoid both non-occupational and occupational noise exposure for 14 hours prior to audiometric testing.

Audiograms will be compared to baseline and prior tests to determine if a standard threshold shift has occurred. If a shift is detected, retesting may be done within 30 days. If a shift is confirmed, the employee will be informed in writing and may need to be refitted and retrained in hearing protection use. If subsequent testing shows that a standard threshold shift is not present, the employee will be informed. Additional audiometric testing may be conducted at the discretion of Health and Safety.

The Physician or audiologist will determine if further evaluation is needed and, if so, will provide to the specialist all the information that is required by 29 CFR 1910.95 (g)(7)(iii). If the physician determines that the medical pathology is unrelated to work exposure or wearing hearing protectors, the employee will be informed by the physician.

#### 5.3 Hearing Protection Devices

Employees must use hearing protection selected, supplies, and/or approved by the firm. Requests for hearing protective devices must be directed to the Regional Health and Safety Managers and Specialists. Hearing protective devices are required to be selected by the Project Manager, or their designee, based on consultation with Corporate HS staff.

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#### 6. TRAINING

Employees required to wear hearing protection will receive training as provided by Corporate H&S. The training will be provided at least annually with refresher training as necessary and will include information regarding: effects of noise on hearing, the purpose of hearing protectors, their advantages/disadvantages and attenuation of various types, the proper selection, fit, use and care of protectors, and the purpose of audiometric testing. Employees will be trained concerning site specific noises hazards and hearing protection by H&S or project H&S staff as applicable.

#### 7. REFERENCES

• OSHA Standard 29 CFR 1910.95

#### 8. RECORDS

**Record Maintenance** – All records regarding noise exposure measurements will be maintained by the offices for two years. All audiometric test records will be maintained for the duration of the affected employee's employment.

#### 9. APPROVALS AND HISTORY OF CHANGE

Approved By:

Mija A. Coppola, Director, H&S Compliance Assurance, LPS, Communications

Revis

Number

**Reason for change** 

1 December 2007	01	Original document
30 January 2008	02	Change to new template

# **ARCADIS**

### Appendix H

Emergency Action Plan and Route to Hospital



## **EMERGENCY ACTION PLAN**

## **Emergency Contact List**

Emergency Contact	Phone
Local Police – New York City Police Department (60 <sup>th</sup> Precinct)	911 (if appropriate) and 212.334.0611
Local Ambulance – New York City Fire Department	911
Local Fire Department – New York City Fire Department	911
Local Hospital – Coney Island Hospital	718.616.3000
Local Weather Data – John F. Kennedy International Airport	718.244.4444
Poison Control	800.332.3073
National Response Center (all spills in reportable quantities)	800.424.8802
U.S. Coast Guard (spills to water)	800.424.8802
Project Manager – Steven Feldman	Office: 631.391.5244 Cell: 516.369.6609
Site Manager – TBD	
H&S Manager – Charles Webster	Office: 315.671.9297 Cell: 315.247.5971
Client Contact – Andrew Prophete	Office: 718.963.5412 Cell: 516.790.1654

# **ARCADIS**

List the Emergency Notification Procedure for the project:

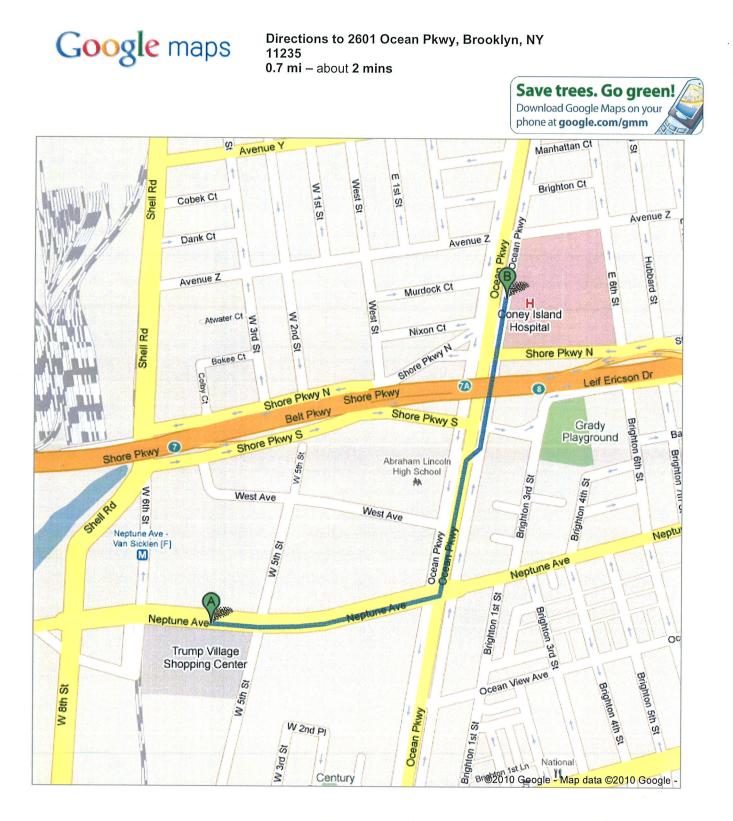
Step 1: Call 911 Step 2: Obtain proper medical care Step 3: Report incident to Project Manager Step 4: Report incident to Work Care

If emergency attention is not needed but professional medical attention is necessary, the employee will be taken to (see hospital route):

Medical Facility:	Coney Island Hospital
Address:	2601 Ocean Parkway
	Brooklyn, NY 11235
Phone Number:	(718) 616-3000

## **Emergency Supplies and Equipment List**

Emergency Supplies and Equipment (check all that apply)	Location on Project Site
🛛 First Aid Kit	Field Vehicles
Sire Extinguisher	Field Vehicles
Mobile Phone Satellite Phone	All Field Personnel
⊠ Traffic Cones	Field Vehicles
U Walkie Talkies	
Water or Other Fluid Replenishment	Field Vehicles
Eye Wash/Quick Drench Station	
🖾 Eye Wash Bottle	Field Vehicles
☑ Wash and Dry Towelettes	Field Vehicles
Sunscreen (SPF 15 or higher)	Field Vehicles
☑ Insect Repellant	Field Vehicles
🛛 Chemical Spill Kit	Field Vehicles
Other (specify):	



<ol> <li>Head east on Neptune Ave toward W 5th St</li> </ol>	go 0.3 m total 0.3 m
2. Turn left at Ocean Pkwy About 1 min	go 0.2 m total 0.5 m
3. Take the 1st <b>right</b> toward <b>Ocean Pkwy</b>	go 121 f total 0.5 m
<ol> <li>Slight left at Ocean Pkwy Destination will be on the right About 1 min</li> </ol>	go 0.2 m total 0.7 m

These directions are for planning purposes only. You may find that construction projects, traffic, weather, or other events may cause conditions to differ from the map results, and you should plan your route accordingly. You must obey all signs or notices regarding your route.

Map data ©2010 Google

Directions weren't right? Please find your route on maps.google.com and click "Report a problem" at the bottom left.

# **ARCADIS**

## Appendix F

Citizen Participation Plan

# **CITIZEN PARTICIPATION PLAN**

## FOR THE

## FORMER DANGMAN PARK MANUFACTURED GAS PLANT SITE

Neptune Avenue and West 5<sup>th</sup> Street Brooklyn (Kings County), NY

Prepared by

National Grid September 2011

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#### **1.0 INTRODUCTION**

Citizen participation is an integral component of remedial programs in New York State. Input from affected or interested individuals and organizations on the remedial program helps ensure outcomes that account for both technical and human concerns for protecting public health and the environment. A project-specific plan is needed to inform and involve community residents, public and private leaders, and other stakeholders. This Citizen Participation Plan (CPP) documents the planned project-specific public outreach activities and resources organized for the remedial program associated with the former Dangman Park Site (Site), a former Manufactured Gas Plant (MGP) Site.

The primary purpose of this CPP is to outline a variety of communication methods that, based on applicable New York State law and New York State Department of Environmental Conservation (NYSDEC) regulations and guidance, provide for constructive communication of program activities between the stakeholders and other interested parties. This CPP includes methods intended to inform interested parties of program developments, elicit responses and public involvement, and provide a central point of contact for inquiries regarding the remedial program for the Site. Given this context, this CPP presents the planned communication and outreach activities, describes how interested individuals and groups can participate in the remedial program, and provides a variety of reference materials to facilitate gaining access to project-specific information and management personnel.

Both the NYSDEC and National Grid are committed to the implementation of this CPP as required by Environmental Conservation Law 27-1417 and Title 6 of the New York Codes, Rules and Regulations (NYCRR) Part 375, applicable NYSDEC guidance (e.g., DER-23/Citizen Participation Handbook for Remedial Programs (January 2010) and the Order on Consent (Index #A2-0552-0606). As required by 6 NYCRR Part 375-1.10 and 375-3.10, NYSDEC and National Grid will review and update this CPP to account for significant changes in the remedial program.

#### 2.0 BASIC SITE INFORMATION

The Dangman Park former Manufactured Gas Plant (MGP) Site (the Site) is located at 486 Neptune Avenue in the Brooklyn, Kings County, New York. The Site is located approximately 1,300 feet southeast of Coney Island Creek and approximately 2,400 feet north of New York Bay. The Site is located in the Coney Island neighborhood of Brooklyn on approximately 1 acre of land, and is contained within Lots 1 and 25 of Block 7273, which are bounded by Neptune Avenue to the north, W. 5<sup>th</sup> Street to the east, a residential parcel to the south and a commercial parcel to the west. Currently, the Site is developed with a shopping center and its parking areas as well as a parking lot for an adjacent high-rise apartment building.

#### History and Operations

Gas used for cooking, lighting, heating and commercial purposes was manufactured at the Site. The Site was operated by the Brooklyn Borough Gas Company, which was a predecessor company to National Grid. The Brooklyn Borough Gas Company was acquired by the Brooklyn Union Gas Company (BUG), which ultimately became KeySpan Corporation (KeySpan). KeySpan became National Grid following a merger in 2008. Based on a review of available historical information, the Site was used as an MGP from prior to 1895 until sometime between 1906 and 1930. The 1895 Sanborn map shows two gas holders, a retort house, two oil tanks, a tar tank, an engine room, a purifying house and a shed. By 1906, the MGP Site was operated by the Brooklyn Borough Gas Company; an additional gas holder, generating house and cistern had been constructed, and the retort house and tar tank were no longer present. The MGP structures were removed sometime between 1906 and 1930. By 1930, the Site was occupied by a club house. By 1966, the Trump Village Shopping Center occupied the northern and central portions of the Site.

#### Current Site Description

As noted, the Site is developed with a shopping center and its parking areas as well as a parking lot for an adjacent high-rise apartment building.



Aerial Site Map of Dangman Park Former MGP Site

#### 3.0 Remedial Program Overview

#### 3.1 New York State Remedial Program Overview

National Grid, successor to KeySpan, conducted a Site Characterization at the Site in 2009 and 2010. The Site Characterization was completed under the 2007 Multi-Site Administrative Order on Consent (ACO) #A2-0552-0606 between KeySpan (now National Grid) and the NYSDEC.

For more information on the remedial program and process in New York State, interested parties can contact any of the state representatives listed in Appendix D or visit the NYSDEC Website at: http://www.dec.ny.gov/chemical/8430.html.

#### 3.2 **Project Area Investigation History**

National Grid conducted a Site Characterization in 2009 and 2010. The Site Characterization was conducted through analysis of samples taken from near-surface (2 to 3 feet below land surface) and deeper soils, groundwater, sub-slab soil vapor and indoor air. A Site Characterization Data Summary report was submitted to the NYSDEC in April 2010 and a Site Characterization Data Summary Addendum report was submitted to the NYSDEC in May 2010. The Key Findings of the Site Characterization are:

- The primary volatile organic compounds (VOCs) that were detected in soil include benzene, toluene, ethylbenzene, and xylenes (BTEX). The primary semi-volatile organic compounds (SVOCs) that were detected in soil include polycyclic aromatic hydrocarbons (PAHs). These compounds are frequently associated with former MGP operations. Three (3) metals (manganese, selenium, and mercury) were detected in soil. These metals may be associated with former MGP operations, but may be present at the Site due to the post-MGP placement of fill material. Total cyanide was detected in soil in four soil samples; as noted above, cyanide may be associated with the post-MGP placement of impacted fill material.
- The primary VOCs and SVOCs that were detected in groundwater include BTEX and PAHs, respectively. Neither light non-aqueous phase liquid (LNAPL) nor DNAPL were detected in any of the monitoring wells.
- The hydrocarbon product identification data for soil samples suggest that fuel oil impacts of unknown origin are present at the water table across the entire area that the former MGP occupied and that coal tar impacts are present at the water table across the central and eastern portion of the area that the former MGP occupied.

- The former gas holders, tar tank, and cistern are all likely sources of the tar releases from the former MGP. Tar-saturated soils were observed in the glacial outwash deposits underlying the Site.
- The highest BTEX and PAH concentrations in soil generally correspond with the observed tar and petroleum impacts, which are a continuing source of groundwater impacts.
- The extent to which elevated concentrations of BTEX and light-end PAH compounds in groundwater have migrated along the groundwater flow path (northwest) is unknown.
- Further investigation is required to characterize the distribution of MGP residuals on Lot 1 and the adjacent parcel to the south (Lot 25), and to delineate the lateral and vertical extent of groundwater impacts downgradient (i.e., northwest) of the former MGP.
- Potential MGP-related constituent vapors are not migrating into the shopping center building at concentrations that may result in an unacceptable human health risk. This is evidenced by the fact that potential MGP-related constituents detected in indoor air were below typical background indoor air concentrations for all indoor air quality samples. Furthermore, the potential MGP-related constituents detected in indoor air may be attributable to other sources (i.e., background sources).
- Tetrachloroethene (PCE) and other chlorinated VOCs (trichloroethene (TCE), cis-1,2dichloroethene, and vinyl chloride) that are not associated with former MGP operations were detected at elevated concentrations in one sub-slab soil vapor sample. A number of non-MGP-related constituents detected in indoor air (2-butanone [methyl ethyl ketone], dichlorodifluoromethane (Freon 12), 4-methyl-2-pentanone [MIBK], and PCE) were above typical background indoor air concentrations in a number of indoor air quality samples.

#### 4.0 CITIZEN PARTICIPATION ACTIVITIES

This section presents the specific citizen participation and outreach activities planned for implementation during the remedial program and to be implemented in accordance with 6 NYCRR Part 375 and DER-23. Operating under project-specific citizen participation goals, clearly defined objectives will be achieved by implementing a range of communication tools and methods. The planned activities are geared toward making project-specific information (e.g., work plans, technical reports, information sheet summaries) available to the public; facilitating communication among stakeholders including the creation of contact lists; scheduling and conducting public meetings; establishing comment periods; and notifying the public of document availability, public meetings, comment periods and major program milestones.

National Grid and the NYSDEC have maintained a limited communications presence in the community near the site throughout the early phases of the project. These activities included briefings of local officials and other interested parties and the publication and distribution of Fact Sheets announcing the Site Characterization. A Fact Sheet has been prepared to announce the start of the Remedial Investigation.

#### 4.1 Goals and Objectives

The central goal of this CPP is to achieve effective, open communication about the Remedial Design and remedial construction among stakeholders and interested parties, National Grid and the NYSDEC. Common goals include:

- Communicate program goals and major milestones, actions and outcomes;
- Inform citizens and others of ongoing project activities, status and progress;
- Provide citizens (and all stakeholders) a forum for input and comment; and
- Engender a public understanding of constituents of interest, their potential effects on human health and the environment, and appropriate responses to mitigate those effects.

In order to accomplish these goals, the following specific objectives will be pursued through the implementation of this CPP:

- Consistently communicate goals, accomplishments and status of the project to the contact list (including community leaders, public officials and the wider community, as necessary) through appropriate means;
- Establish, maintain, update and utilize the contact lists;
- Educate the community, in lay terms, about the nature and magnitude of potential site risks, including instructions for mitigating risk (if appropriate) and assurances that the environment and worker/public health and safety are protected;
- Provide interested parties the opportunity to review and comment on technical reports generated through the remedial program (e.g., public comment periods and document repository as required by 6 NYCRR Part 375);
- Provide interested parties the opportunity to present opinions and ideas during the remedial program (e.g., conduct public meeting/comment period and availability session as required by 6 NYCRR Part 375);
- Provide the news media with interviews of National Grid authorized spokespersons, as available, as well as press releases and similar materials to ensure accurate coverage of remedial program activities;
- Provide a designated project spokesperson as point of contact through which community inquiries regarding the project can be addressed consistently and effectively; and
- Periodically review the effectiveness of the citizen participation and outreach activities during the remedial program and make adjustments in this CPP's methods and/or activities, if necessary.

The community contact list is provided in Appendix C and the former Dangman Park MGP Site Project Management contacts (NYSDEC, NYSDOH and National Grid representatives) are provided in Appendix D.

#### 4.2 Tools and Methods

There are many ways to reach and communicate with the community and other interested parties as this CPP is implemented over the course of the remedial program. A variety of outreach tools and methods will be used to ensure proper communication with the interested parties that include various organizations, public and business leaders, and a diverse assemblage of individuals of all ages, education backgrounds and cultures.

Interested parties will be informed and invited to participate in the planned citizen participation activities through appropriate means such as mailings to the contact list, legal notice in newspapers, press releases, notices posted on the site's website, information sheets and other documents made available in the document repository.

The following specific public participation activities will be implemented as required by 6 NYCRR Part 375 and current NYSDEC guidance (DER-23).

### 4.2.1 Document Repository

A Local Document Repository has been established at the offices of Brooklyn Community Board 13, which maintain in one file all of the relevant documents related to the Site. A Repository is also maintained at the NYSDEC offices in Albany, New York. Contact information for the Local Document Repository:

Brooklyn Community Board 13 1201 Surf Avenue Brooklyn NY 11224 718 266 3001 Attn.: Chuck Reichenthal

The following documents are available in the Repository, or will be made available when completed and accepted by the NYSDEC:

- Citizen Participation Plan
- Site Characterization Data Summary and Site Characterization Data Summary Addendum
- Remedial Investigation Work Plans (including Supplemental Investigations)
- Final Remedial Investigation Report
- Feasibility Study

- Final Remedial Design Work Plan
- Remedial Action Work Plan
- Remedial Design
- Construction Completion Report
- Site Management Plan
- Other Materials (e.g., Information Sheets, Notices, etc.).

#### 4.2.2 Public Meetings and Fact Sheets

Milestone Public Meetings will be scheduled by the NYSDEC and Fact Sheets announcing such meetings will be distributed to the site's mailing list. Pursuant to DER-23, a Public Meeting will be scheduled at the time of Proposed Remedial Action Plan. Fact Sheets will be produced and distributed to the Site's contact list at the time the NYSDEC issues a Record of Decision, before the start of Remedial Action (after the acceptance of the Remedial Design Report) and at the issuance of the Certificate of Completion. During the construction period, National Grid, in consultation with the Site owners, will prepare for NYSDEC review and distribution a quarterly Fact Sheet summarizing construction progress.

#### 4.2.3 Information Newsletters/Handouts

Information newsletters will be prepared and distributed to the contact list in order to announce activities that may impact normal community activities or to announce major project milestones and accomplishments throughout the remedial program (e.g., start of construction, major report completion, etc.). Written in lay terms, information newsletters or handouts will describe and summarize current and planned work on the Site, particularly any which may impact routine community activities.

#### 4.2.4. <u>Telephone Hotline</u>

National Grid has established a Telephone Hotline for neighbors of the Site. The phone number is (718) 403-3014. There will be occasional news updates about the investigation, and the Hotline can be used at any time to leave questions about the project.

#### 4.2.5 Website

National Grid is establishing a website for the project at <u>www.dangmanparkmgpsite.com</u>. The website will include background information about manufactured gas plants, historical information about the Site, a description of work that has been completed during the project and contact information, including links to the NYSDEC's MGP website. The home page will contain a periodic update of activities related to the investigation and remediation of the Site. A Key Documents section will contain all of the major reports completed during the project, and an archive of Fact Sheets and weekly reports.

#### 4.3 Roles and Responsibilities

The specific roles and associated responsibilities for implementing this CPP are:

- NYSDEC Remedial Project Manager The NYSDEC Project Manager is responsible for enforcement, oversight and management of the overall remedial program. Typical citizen participation-related activities include making presentations at public meetings, reviewing project documents such as information sheets and providing technical assistance in preparing the responsiveness summary or answering public inquiries.
- NYSDEC Citizen Participation Specialist The Citizen Participation Specialist assists the project manager in implementing the CPP. Typical activities include preparation and/or review of information sheets and the responsiveness summary and coordination of public meetings and availability sessions.
- National Grid Project Manager The National Grid Project Manager, in cooperation with the NYSDEC Project Manager, is responsible for implementing the overall remedial program at the site. Typical citizen participation-related activities include management of CPP implementation, presentations at public meetings and technical assistance to the NYSDEC Project Manager and Citizen Participation Specialist.

#### 4.4 Schedule for Implementing Elements of the CPP

Implementing elements of this CPP will depend upon completion by National Grid and final approval by the NYSDEC of various plans and reports required by the Order on Consent governing the Site.

#### 5.0 SUMMARY

Guided by the goals and objectives of this CPP, implementation of the planned public outreach and citizen participation activities will ensure the timely communication of important program information of interest to the local community. Citizen involvement and interaction in the remedial program will be facilitated through specific opportunities such as public meetings, public comment periods, availability sessions and use of the Site's website and the Document Repository. Throughout the remedial program, this CPP and its specific outreach tools and methods will be monitored and, as required and agreed by the NYSDEC and National Grid will be adjusted to improve its effectiveness in responding to community needs.

## Appendix A

### GLOSSARY OF KEY CITIZEN PARTICIPATION TERMS AND MAJOR PROGRAM ELEMENTS

**Citizen Participation Plan (CPP)** - A document that describes the project-specific citizen participation and outreach activities that will take place alongside the technical components of the remedial program. The CPP also provides project information, citizen participation goals and objectives, and lists of contact persons and document repositories.

**Citizen Participation Specialist** - An NYSDEC staff member whose duty it is to provide guidance and assistance in carrying out the CPP. The Citizen Participation Specialist is the key contact for public inquiries about the project and the remedial activities.

**Contact List** - A list in the CPP (Appendix C) containing names and addresses of individuals, groups, organizations, news media and public representatives interested and/or affected by the project. The contact list is used to distribute important information and notices about the project and the remedial program.

**Document Repository** - Project documents and other information are placed in the Document Repository to facilitate convenient public access to these materials. Documents are available for public reference and review at the offices of Community Board 13. Refer to Appendix B for more information about the Document Repository location.

**Feasibility Study (FS)** - Based on information gathered during the Remedial Investigation (RI), the FS is a process for developing, evaluating and selecting appropriate Remedial Action (RAs) for limiting or eliminating the potential human and environmental hazards of a site. The FS sets out the goals of the remedial actions to be taken, evaluates the most appropriate alternatives and selects the best alternative based on several criteria. The selected remedy is then recommended for implementation in the Proposed Remedial Action Plan (PRAP), which is subject to public review and comment.

**Interim Remedial Measure (IRM) - A** discrete action which can be conducted at a site relatively quickly to reduce the risk to people's health and the environment from a well defined waste problem. An IRM can involve cutting and plugging waste conduits, removing contaminated soil and securing a site.

**Polycyclic Aromatic Hydrocarbons** (PAHs) - Contaminants typically found at MGP sites and associated with coal tar residues.

**Remedial Design (RD)** - This report will include a detailed description of the remedial objectives and the means by which each essential element of the selected remedial alternative will be implemented to achieve those objectives. It incorporates the findings of the FS Report to provide a remedial design which will be implemented during the performance of the cleanup activities at the site.

**Remedial Investigation** (**RI**) - A process to determine the nature and extent of contamination at a site by analyzing data collected from sampling (e.g., water, soil, air, etc.) at a site. Information gathered throughout the RI is then used to conduct a Feasibility Study (FS), which proposes and evaluates various remedial alternatives for the site.

**Responsiveness Summary** - The Responsiveness Summary is prepared by the NYSDEC to address public comments, questions and concerns regarding the proposed remedial action (PRAP) to be taken at a site. The Responsiveness Summary is issued as part of the Remedial Action Program.

**Volatile Aromatic Hydrocarbons** - Benzene, Toluene, Ethylbenzene, Xylene (BTEX) - Volatile aromatic hydrocarbons and are typical contaminants found at MGP sites and other sites where coal, oil, refined products and other hydrocarbons were burned or used.

## Appendix B

## **IDENTIFICATION OF DOCUMENT REPOSITORIES**

Community Board 13 1201 Surf Avenue Brooklyn NY 11224 718 266 3001 Attn.: Chuck Reichenthal -- Call for Appointment

NYSDEC Division of Environmental Remediation 625 Broadway Albany, NY 12233-7014 (518) 402-9564 Call for Appointment and Hours

# Appendix C

## **IDENTIFICATION OF AFFECTED AND/OR INTERESTED PARTIES**

## Site Owners:

Trump Village Construction Corporation Sheepshead Bay Road Brooklyn, NY 11224 (718) 266-0500

<u>Government Officials</u> <u>Elected Official</u>	<u>Name, address, phone, e-mail</u>
Brooklyn Borough President	Marty Markowitz Brooklyn Borough Hall 209 Joralemon Street Brooklyn, New York 11201 (718) 802-3700
Assemblyman Alec Brook-Krasny brookka@assembly.state.ny.us	2823 West 12th Street Suite 1F Brooklyn, NY 11224 TEL. 718-266-0267
Councilman Dominic Recchia drecchia@council.nyc.gov	445 Neptune Ave. Brooklyn, NY 11224 TEL. 718-373-9673
Congressman Jerrold Nadler www.house.gov/nadler	445 Neptune Ave. (District Office) Brooklyn, NY 11224 TEL. 718-373-3198 FAX 718-996-0039
NYS Senator Carl Kruger kruger@senate.state.ny.us	2201 Avenue U Brooklyn, NY 11229

TEL. 718-743-8610

## **Community Groups**

**Community Board** 

Community Board 13 Chuck Reichenthal, District Manager 2900 West 8<sup>th</sup> Street Brooklyn NY 11224 718 266 3001

## **Community Newspapers**

<u>Newspaper Name</u>	<b>Editor's Name</b>	Address, Phone #, E-mail
Brooklyn Papers	Gersh Kuntzman	(718) 834-9350, gersh.kuntzman@verizon.net
Brooklyn Eagle	Henrik Krogius	(718) 858-7474 <u>kgrogius@brooklyneagle.net</u>
Bay Currents		2966 Avenue U, Suite 108 Brooklyn, NY 11229 347-492-4432 www.baycurrents.net

## Appendix D

#### **IDENTIFICATION OF PROJECT MANAGEMENT CONTACTS**

#### New York State Department of Environmental Conservation

Hank Willems Project Manager NYSDEC Division of Environmental Remediation 625 Broadway Albany, NY 12233-7017 (518) 402-9662

#### New York State Department of Health

Albert DeMarco NYSDOH Bureau of Environmental Exposure Investigation 547 River Street Troy, NY 12180-2216 (518) 402-7880, or 1-(800) 458-1158

#### National Grid Public Representative

April Dubison Community Relations National Grid Fleet Services Administration Building 287 Maspeth Ave Brooklyn, NY 11211

Dangman Park MGP Project Hotline Telephone - (718) 403-3014

Please leave a message on the Hotline and your call will be promptly returned.