



## **Vapor Intrusion Verification Work Plan**

### **Motors Liquidation Company**

Former Delphi Harrison Thermal Systems Moraine Plant  
Former General Motors Powertrain Group, Moraine Engine Plant  
Former General Motors Truck Group, Moraine Assembly Plant

Moraine, Ohio

March 11, 2010

Revised June 21, 2010

Revised August 20, 2010

Revised September 16, 2010

<b>1. Introduction</b>	<b>1</b>
1.1 Site Background	2
1.2 Baseline Risk Assessment Summary	2
1.3 Vapor Intrusion Verification Work Plan Objectives	3
1.4 Vapor Intrusion	4
<b>2. Vapor Intrusion Study within Neighborhood Southwest of the Site</b>	<b>6</b>
2.1 Groundwater Sampling Results	6
2.2 Groundwater and Soil-Gas Sampling Points	7
<b>3. Vapor Intrusion Study East of the Site</b>	<b>9</b>
3.1 Groundwater Sampling Results	9
3.2 Groundwater and Soil-Gas Sampling Points	10
<b>4. Soil Gas Sampling Point Installation</b>	<b>11</b>
4.1 Drilling Methodology	11
4.2 Groundwater Sample Collection	11
4.3 Historic Groundwater Table Fluctuations	12
4.4 Soil-Gas Sample Point Installation	12
<b>5. Soil-Gas Sampling</b>	<b>14</b>
<b>6. Schedule</b>	<b>15</b>
<b>7. Reporting</b>	<b>16</b>
<b>8. References</b>	<b>17</b>

**Tables**

- 1 Property Information Details for Neighborhood Located Southwest of Site.
- 2 Recent Groundwater Sampling Data and Vertical Aquifer Profiling Data from Select Monitoring Wells.
- 3 Historic Groundwater Table Fluctuations.
- 4 Generic Screening Levels for Soil-Gas.

**Figures**

- 1 Site Layout.
- 2 Aerial Photograph View of Neighborhood to Southwest of Site.
- 3 Aerial Photograph View East of Site.
- 4 Cross Section A-A'.
- 5 Cross Section B-B'.
- 6 Soil-Gas Nested and Temporary Groundwater Sampling Points.
- 7 Groundwater Elevation Fluctuations Southwest Neighborhood (2006 through 2009).
- 8 Groundwater Elevation Fluctuations Eastern Portion of Site (2006 through 2009).
- 9 Deepest Soil-Gas Point Screen Location Decision Matrix.
- 10 Schedule for Vapor Intrusion Verification Implementation.

**Appendices**

- A Standard Operating Procedures
- B Laboratory Methodology and QA/QC
- C Selected Boring Logs

## 1. Introduction

As discussed during a meeting on February 11, 2010 between the United States Environmental Protection Agency (U.S. EPA) and Motors Liquidation Company (MLC), a field investigation was proposed to provide site-specific data to verify the previously completed vapor intrusion risk assessment. This field investigation is being completed to provide one line of evidence to further evaluate the potential for the vapor intrusion pathway using a phased approach. During this field investigation soil-gas and groundwater table samples will be collected. The field investigation will be focused near potential off-site receptors (residential structures) located within proximity of the MLC (formerly General Motors Corporation [GM]) former Moraine Facilities located in Moraine, Ohio (Site). The areas of the field investigation are the neighborhoods located to the southwest of the Site (Figures 1 and 2) and east of the Site (Figures 1 and 3). For both areas, the groundwater and soil vapor data will be used as part of the overall weight of evidence for evaluating the vapor intrusion pathway. The specific data and results to be considered for each area are presented below.

- Vapor Intrusion Study Southwest of the Site: Based on the existing lines of evidence 1) groundwater in the upper aquifer exhibits concentrations of site-specific volatile organic compound (VOCs) that exceed Maximum Contaminant Levels (MCLs) and 2) the risk assessment concluded that current and reasonably expected future exposure of off-site residents to groundwater via vapor intrusion is not expected to be significant. Due to the presence of VOCs in groundwater potentially serving as a source of a vapor phase plume, additional verification sampling in the neighborhood to the southwest (indoor air and sub slab samples) will be conducted in a future phase regardless of the findings of this investigation.
- Vapor Intrusion Study East of the Site: Based on the existing lines of evidence 1) groundwater in the upper aquifer does not contain site-specific VOC concentrations that exceed MCLs; 2) residential homes are located greater than 100 feet from groundwater containing VOCs above the MCL; 3) waste management units were not identified on the east side of the former Moraine facilities; and 4) the risk assessment concluded that current and reasonably expected future exposure of off-site residents to groundwater via vapor intrusion is not expected to be significant. Based on the lack of a VOC source in soil or groundwater, a future phase of investigation to the east will likely not be warranted.

This Vapor Intrusion Verification Work Plan (Work Plan) presents the scope of work for the collection of soil-gas samples in these investigation areas.

## 1.1 Site Background

A multi-phased Resource Conservation and Recovery Act (RCRA) Facility Investigation (RFI) was completed for the following former Moraine Facilities Sites (Figure 1): former Delphi Harrison Thermal Systems Moraine Plant (former Delphi Thermal Moraine [leased to Delphi Corporation between January 1999 and September 2003]), former General Motors Powertrain Group, Moraine Engine Plant (former Moraine Engine), and former General Motors Truck Group, Moraine Assembly Plant (former Moraine Assembly) and approved by the U.S. EPA in June 2000 (ARCADIS Geraghty & Miller, Inc. 2000a and b, ENVIRON Corporation 2000a and b). The RFI, conducted during the period of 1992 to 1998, identified that the primary source area of VOCs at the three former facilities is located at the Area of Interest (AOI) 7 - Former Oil House Area, in the northern portion of the former Moraine Engine facility. Former GM implemented a supplemental groundwater investigation from 2005 to 2008 to further refine the understanding of groundwater chemistry and hydrogeology both on-site and off-site for the final corrective measures implementation. Interim Measures to address the VOCs in groundwater have been implemented since 1996 for the upper aquifer and 1991 for the lower aquifer. The site-specific VOC parameter list is: benzene, 1,1-dichloroethane (1,1-DCA), 1,1-dichloroethene (1,1-DCE), cis-1,2-DCE, trans-1,2-dichloroethene (trans-1,2-DCE), ethylbenzene, tetrachloroethene (PCE), toluene, 1,1,1-trichloroethane (1,1,1-TCA), trichloroethene (TCE), vinyl chloride, and xylenes. The site conceptual model, the updated human health risk assessment, the interim measures completed to date, and the final recommended site-wide corrective measures were presented in the Corrective Measures Proposal (CMP) (ARCADIS, Inc. 2008).

## 1.2 Baseline Risk Assessment Summary

As part of the CMP (ARCADIS, Inc. 2008), an updated evaluation including Johnson and Ettinger (J&E) modeling was completed for potential soil and groundwater exposure pathways and receptors identified since the completion of the RFI (ENVIRON Corporation 2000a and b). Included in this evaluation was exposure of on-site indoor workers to constituents in soil and groundwater via vapor intrusion and exposure of off-site routine workers and residents to constituents in groundwater via vapor intrusion. Details of this evaluation are located in Appendix G "Supplemental Human Health Risk Assessment" of the CMP.

The conclusions of the updated risk analysis are presented in the CMP and are summarized as follows:

#### Routine Worker Vapor Intrusion Exposure

The risk assessment completed as part of the CMP evaluated potential exposure of on- and off-site routine workers to groundwater via vapor intrusion. Cumulative cancer risk and hazard index (HI) estimates were computed for hypothetical routine worker vapor intrusion exposure to groundwater at and downgradient of the Site. Based on sampling data from on- and off-site monitoring wells the well-specific exposure estimates did not exceed a cumulative cancer risk limit of  $10^{-4}$  or non-cancer HI limit of 1 for any on-site or off-site wells. Therefore, current and reasonably expected future exposure of on- or off-site routine workers to groundwater via vapor intrusion is not expected to be significant.

#### Resident Vapor Intrusion Exposure

The risk assessment completed as part of the CMP evaluated potential exposure of off-site residents to groundwater via vapor intrusion. Based on data obtained from on- and off-site monitoring wells, well-by-well cancer risk and non-cancer HI estimates for resident groundwater vapor intrusion exposure do not exceed the cumulative cancer risk limit of  $10^{-4}$  or the HI limit of 1 for any on-site and off-site wells. Therefore, current and reasonably expected future exposure of off-site residents to groundwater via vapor intrusion is not expected to be significant.

### **1.3 Vapor Intrusion Verification Work Plan Objectives**

The overall objective of this Work Plan is to collect additional lines of evidence for evaluating the vapor intrusion pathway. The specific objectives for the southwest and east areas are as follows:

- Evaluate the presence of site-specific VOCs in soil-gas in the neighborhood right-of-way southwest and east of the Site.
- Identify VOC concentrations in soil-gas at each of the sampling depths as a means to determine soil-gas migration and attenuation moving away from source areas (i.e., groundwater). Soil-gas data will also be used to correlate VOC concentrations below nearby residential homes and aid in the selection of homes for additional sampling southwest of the site.
- Evaluate the potential for groundwater to serve as a source for VOCs in soil-gas. This will be accomplished by collecting water table groundwater samples from temporary well points at the same location and prior to installation of the soil-gas

sampling points to qualitatively determine concentrations of site-specific VOCs within shallow groundwater (presence or absence).

- Update the site conceptual model through the collection of site-specific geotechnical data.
- Confirm concentrations of site-specific VOCs in groundwater remain consistent with historic sampling data east of the Site in permanent monitoring wells GM-25 and GM-77S (quantitative data).

#### **1.4 Vapor Intrusion**

Vapor intrusion is defined as vapor phase migration of volatile organic and/or inorganic compounds into occupied buildings from underlying contaminated groundwater and/or soil. A vapor intrusion pathway is complete if there is human exposure to the vapors. If the pathway is complete, the vapor concentrations should be assessed to determine if the potential risk to human health is acceptable or unacceptable. If the risk is unacceptable then vapor mitigation may be implemented. There are multiple ways to evaluate vapor intrusion. The three basic types of evaluation by physical sampling include soil-gas, sub-slab gas, and indoor air sampling. Soil-gas sampling is generally used to focus a subsequent sub-slab or indoor air investigation; however, it also provides an additional line of evidence to further understand the possibility that the pathway is incomplete. Sub-slab sampling is a multiple step process that requires property access and coordination with the building owner and/or occupant to core through the building foundation and collect an air sample. Indoor air sampling is the collection of vapor samples within a building. Indoor air sampling may have complications such as the potential for background sources of vapors (e.g., household cleaners, gasoline).

Soil-gas sampling will be used at the Site investigation areas to evaluate whether VOCs are present in soil-gas and to evaluate soil-gas migration and attenuation moving away from the source (i.e., groundwater). Nested multi-depth ports will be installed to determine a vertical profile and calculate attenuation factors for vapors migrating through soil. Soil-gas sampling points installed near the groundwater table will be used to evaluate the partitioning of constituents from groundwater to the vapor phase (for soil-gas samples collected near monitoring wells). Collecting soil-gas samples from immediately above the groundwater which may be serving as a source should be a worst-case scenario. Shallow soil-gas sampling points will be installed at depths just below the depth of a typical basement and slab-on-grade foundation.

Additionally, soil-gas data, along with site-specific geotechnical data, may be used to refine the site conceptual model regarding the vapor intrusion pathway.

## 2. Vapor Intrusion Study within Neighborhood Southwest of the Site

The neighborhood located to the southwest of the Site consists of approximately 60 occupied structures on approximately 75 residential parcels. Additional properties including a church located at 3001 Lakehurst Court, and an active gas station/service shop located at 2901 Sellars/Main Street are present within this neighborhood.

The active gas station/service shop located at 2901 Sellars/Main Street contains underground storage tanks (USTs). All UST facilities must register and abide by all regulations and laws under the Bureau of Underground Storage Tank Regulations (BUSTR) under the Ohio State Fire Marshall's office. The gas station (Moraine Drive-Thru, Inc.) is registered under facility ID 57000030. The gas station has USTs that contain gasoline, diesel and kerosene; however, no active releases have been reported. The constituents associated with petroleum use under the BUSTR 2005 Technical Guidance Manual fall under Analytical Groups 1 and 2 and included benzene, toluene, ethylbenzene, xylenes (BTEX), polynuclear aromatics, and total petroleum hydrocarbons. BTEX is part of the site-specific VOC list; however, BTEX constituents are not present in the groundwater plume extending beneath the neighborhood to the southwest. If BTEX is detected in soil-gas samples collected in close proximity to the gas station, further evaluation might be required to determine the origin of the soil-gas containing BTEX.

Homes in the neighborhood have partial basements, full basements, crawl-spaces or slab-on-grade construction. Specific construction and property details for this neighborhood are provided on Table 1. To provide an understanding of groundwater quality within the deeper portions of the upper aquifer, Section 2.1 discusses concentrations of site-specific VOCs in groundwater beneath this neighborhood during recent groundwater sampling of permanently installed wells. To verify if shallow groundwater is serving as a source of site-specific VOCs in soil-gas within this neighborhood, soil-gas monitoring points will be installed and shallow groundwater samples will be collected from temporary well points as discussed in Section 2.2.

### 2.1 Groundwater Sampling Results

General groundwater flow is west/southwest in the vicinity of the neighborhood southwest of the Site. Groundwater in the upper aquifer southwest of the Site exhibits concentrations of site-specific VOCs above MCLs.

Recent groundwater sampling results are included on Table 2 and on Figure 4 for site-specific VOCs. Monitoring wells within the neighborhood include well pairs GM-15/16 located south of Old Sellars Road and west of Portage Road, GM-47/50 located on the north side of Telhurst Road and east of Portage Road, and GM-63/64 located north of Hoylake Court between Dryden Road and Portage Road. The most shallow wells of the pairs are GM-16 (well screen is from 48-58 feet below land surface [bls]), GM-50 (well screen is from 30-40 feet bls), and GM-63 (well screen is from 30-40 feet bls). All monitoring wells within the neighborhood are screened below the water table. An additional monitoring well (WSU-23) owned by Wright State University is not associated with the Site. This well is only used for site-wide potentiometric surface mapping.

Detected site-specific VOCs in the upper aquifer wells (GM-16, GM-50, and GM-63) are 1,1,1-Trichloroethane (1,1,1-TCA), 1,1-Dichloroethene (1,1-DCE), Tetrachloroethene (PCE), Trichloroethene (TCE), cis-1,2-Dichloroethene (cis-1,2-DCE), and trans-1,2-DCE. Concentrations detected during the 2009 site-wide groundwater sampling (ARCADIS, Inc. 2010) of PCE and TCE in the shallowest wells were above the U.S. EPA MCL for PCE and TCE. All three wells had concentrations of PCE of 110 micrograms per liter ( $\mu\text{g/L}$ ) and concentrations of TCE ranged from 74  $\mu\text{g/L}$  in GM-16 to 120  $\mu\text{g/L}$  in both GM-50 and GM-63.

## 2.2 Groundwater and Soil-Gas Sampling Points

A total of eight soil-gas sampling points are proposed to be installed within the right-of-way (between the street and the sidewalk) within the neighborhood and one soil-gas sampling point is proposed to be installed across the street near the Moraine City Building (Figure 2). MLC will coordinate access with the City of Moraine and obtain all permits necessary and work with utility companies for clearance to complete the work.

Figure 2 shows the location of the nine soil-gas sampling locations. These locations may be modified based on the presence of utilities. At a minimum, information on utilities will be used to further evaluate soil-gas results. Geotechnical samples will be collected at up to four discreet depth intervals at respective soil-gas sampling locations outlined on Figure 2. Samples will be submitted to DLZ Laboratories in Columbus, Ohio (DLZ) for analysis of grain size (American Society for Testing and Materials [ASTM] Method D422/D1140), specific gravity (ASTM Method D854), permeability (ASTM Method D2434), moisture content (ASTM Method D2216), and air filled/water filled and total porosity (calculation). Details regarding laboratory methods for these analyses and the porosity calculation are included in Appendix B.

Groundwater table samples will also be collected in the same boring as the soil-gas point is installed. A schematic providing details for groundwater sample collection is provided on Figure 6 and methodologies for groundwater sampling using low flow methodology will be discussed in Section 4.2 and Standard Operating Procedure (SOP) 24A in Appendix A.

Methodologies for installation of the soil-gas sampling points will be discussed in Section 4.3 and SOP 36 included in Appendix A. A schematic providing installation details of the soil-gas sampling point is provided on Figure 6. All soil-gas sampling points will be installed as permanent monitor points and completed at the surface with a sub-grade vault with flush-mounted lid and a concrete pad to inhibit tampering and ensure integrity of the soil-gas sampling points. Two soil-gas sampling events will be completed, one event this summer and one event later this fall closer to the heating season. Soil-gas sample collection procedures are presented in Section 5.

### 3. Vapor Intrusion Study East of the Site

East of the Site (along Southbound Kettering Boulevard) is primarily residential. The potential for site-specific VOCs to be present in the soil-gas east of the Site is not expected based on upper aquifer groundwater concentrations from permanently installed wells (Section 3.1). In addition, waste management units were not identified on the east side of the former Moraine facilities. To confirm the absence of site-specific VOCs east of the Site, select upper aquifer groundwater wells will be resampled and three soil-gas monitoring points will be installed and sampled (Section 3.2).

#### 3.1 Groundwater Sampling Results

Groundwater flows west/southwest in vicinity of the eastern boundary of the Site. Concentrations in the upper aquifer groundwater east of the Site exhibit concentrations of site-specific VOCs below MCLs.

Recent groundwater sampling results are included on Table 2. Monitoring wells within the neighborhood east of the Site include well pair GM-77S/77D and GM-84. Concentrations in GM-77S were below detection limits for all site-specific VOCs. GM-77D is screened below the regional clay till. During the 2007/2008 supplemental groundwater investigations, vertical aquifer profile samples were collected at the location of monitoring well GM-84. Concentrations from the shallow upper aquifer sample collected from 32 to 37 feet bls were below laboratory reporting limits for all site-specific VOCs with the exception of PCE (1.4 µg/L). Additional vertical aquifer profile sampling data collected during the installation of monitoring well GM-84 is located on Table 2.

Well pair GM-53/54, and wells GM-58, GM-70, and GM-25 are all located along the eastern boundary of the Site. GM-53 is screened near the water table (well screen from 23-33 feet bls), GM-25 is screened in the lower portion of the upper aquifer (well screen from 48-58 bls), and GM-54, GM-58, and GM-70 are screened in the lower aquifer. Concentrations of site-specific VOCs in upper aquifer wells GM-25 and GM-53 were below detection limits during the most recent sampling data (Table 2).

During the 2006 and 2007/2008 supplemental groundwater investigations, vertical aquifer profile samples were collected at the location of monitoring wells GM-58 and GM-70. The analytical results from a groundwater sample collected from 25-30 feet bls at the location of GM-58 indicated that site-specific VOCs were present above

laboratory reporting levels (ARCADIS, Inc. 2007). The analytical results from a groundwater collected at the location of monitoring well GM-70 from 40-45 feet bls indicated that site-specific VOCs were below reporting limits with the exception of PCE detected with a concentration of 4.5 µg/L (ARCADIS, Inc. 2008).

### 3.2 Groundwater and Soil-Gas Sampling Points

Groundwater samples will be collected from upper aquifer monitoring wells GM-25 and GM-77S. Monitoring well GM-25 was last sampled in 1999 and GM-77S in 2007. Confirming the absence of site-specific VOCs in the groundwater at GM-25 and GM-77S will provide data to confirm that groundwater does not serve as a source of site-specific VOCs in soil-gas.

Three soil-gas sampling points will be installed along the eastern boundary of the Site (Figure 3). The center soil-gas sampling point will be installed east of monitoring well GM-70 on the property boundary with one approximately 350 feet to the northeast along the property boundary and one approximately 500 feet to the southwest along the property boundary (Figure 5). Geotechnical samples will be collected at up to four intervals during installation of the center soil-gas sampling point east of GM-70. Samples will be submitted to DLZ for analysis of grain size (ASTM Method D422/D1140), specific gravity (ASTM Method D854), permeability (ASTM Method D2434), moisture content (ASTM Method D2216), and air filled/water filled and total porosity (calculation). Details regarding laboratory methods for these analyses and the porosity calculation are included in Appendix B.

Groundwater table samples will be collected in the same boring as the soil-gas point is installed. A schematic providing details for groundwater collection is provided on Figure 6 and methodologies for groundwater sampling are discussed in Section 4.2 and SOP 24A in Appendix A.

Methodologies for installation of the soil-gas sampling points are discussed in Section 4.3 and SOP 36 included in Appendix A. A schematic providing installation details of the soil-gas sampling point is provided on Figure 6. The soil-gas sampling points will be considered permanent and completed at the surface with a sub-grade vault with flush-mounted lid and a concrete pad to inhibit tampering and ensure integrity of the soil-gas sampling point. Two soil-gas sampling events will be completed, one event this summer and one event later this fall closer to the heating season. Soil-gas sample collection procedures are presented in Section 5.

## 4. Soil-Gas Sampling Point Installation

### 4.1 Drilling Methodology

The soil-gas sampling points will be drilled and installed using the direct push method. The drill rig will be outfitted with a dual-core sampler to ensure borehole stability during advancement. Prior to drilling, all locations will be cleared of underground utilities by contacting the Ohio Utility Protection Service (OUPS), coordinating with the City of Moraine and Montgomery County and by passive methods such as air- or wet-knifing to a depth of approximately 10 feet bls.

At all locations, the boring will be advanced to the groundwater table. Continuous soil samples will be collected for lithology and screened using a photo-ionization detector (PID). All non-disposable down-hole equipment will be decontaminated between locations.

Geotechnical sample collection at the specified locations will require a separate boring to be completed using hollow-stem auger drilling method. This drilling will be completed by the direct push drilling rig, which is capable of completing borings using hollow-stem auger drilling methods. After completion of these borings, the borehole will be abandoned by using bentonite chips or slurry.

### 4.2 Groundwater Sample Collection

At each of the soil-gas sampling point locations, a water table groundwater sample will be collected from a temporary well point (Figure 6). Once the boring has reached the water table (approximately 20 feet bls), a stainless steel well screen will be extracted from the dual casing (Appendix A). A nominal amount of groundwater will be purged from the well screen using a submersible pump. The groundwater removed during this development will include three times the volume of water within the dual casing. After development, groundwater samples will be collected use low-flow methodology. Additional samples (duplicates – 1 per 20 samples) will be collected and trip blanks added for quality assurance and quality control (QA/QC) requirements. The groundwater samples will be submitted to TestAmerica Laboratories of North Canton, Ohio for analysis of site-specific VOCs by U.S. EPA method 8260B. Laboratory methodology and QA/QC information are attached in electronic format in Appendix B. Details for the groundwater sampling collection are located in Appendix A; SOP 24A. The draft Quality Assurance Project Plan (QAPP) with amendments will be followed for this Work Plan (ARCADIS, Inc. 2003).

### 4.3 Historic Groundwater Table Fluctuations

The soil-gas installation depth nearest to the (groundwater table) will be installed at approximately 2 feet above the maximum groundwater elevation to ensure that the soil-gas sample point screen will not become saturated by groundwater fluctuations. This was determined by examining groundwater levels collected from 2006 through 2009 (Table 3). Groundwater maximum, minimum and mean elevations were calculated from wells in the vicinity of the proposed soil-gas sample point locations. The results from the calculations are presented in Table 3 and graphically presented in Figures 7 and 8 for the neighborhood southwest and the eastern site property boundary, respectively. The groundwater table in the vicinity of the neighborhood southwest of the site fluctuates no more than 5 feet from the minimum to the maximum. The groundwater table at the eastern boundary of the site fluctuates no more than 6 feet from the minimum to the maximum. Setting the soil-gas sampling point screens at approximately 2 feet above the maximum groundwater elevation will ensure fluctuations in groundwater will not compromise the deepest soil-gas sampling point.

### 4.4 Soil-Gas Sample Point Installation

After completion of the boring, three soil-gas sampling points will be installed within each borehole at discreet intervals (Figure 6). The soil-gas sampling points will be installed approximately 2 feet above the maximum groundwater table elevation to collect soil-gas at the potentially highest concentrations present, and at depths to correspond to residential structures with basements and crawl space/slab-on-grade construction. Historic fluctuations of the groundwater table were considered for the determination of the deepest soil-gas sampling point depth. If the current groundwater elevation at the time of installation is higher than the historic maximum groundwater elevation then the deepest point will be adjusted to two feet above the new maximum groundwater elevation. In order to help facilitate this field decision, a decision matrix has been established for setting the deepest soil-gas sampling point (Figure 9). The overall goal is to install the deepest point close to the top of the water table without being submerged (Section 4.3). Based on an analysis of water table depths (see Section 4.3), soil-gas samples will be collected from the following three depths: approximately 2 feet above the maximum groundwater table elevation; 11 feet bls; and 6 feet bls (approximate depth of a basement). Overall, the sample depths were selected to provide information on the vertical attenuation of soil-gas moving away from the groundwater table. The sample at approximately 2 feet above the maximum groundwater table elevation will provide information on VOC concentrations in soil-gas

close to the source and will represent a worst-case scenario. This depth corresponds to approximately 14 to 17-feet bls in the southwest area and 17 to 25 feet bls in the east area, depending on the surface elevation. The 11 foot and 6 foot bls samples will provide information on the attenuation of soil-gas through the soil column. In particular, data from 6 feet bls will provide some preliminary information on concentrations that may be present below nearby residential homes.

After completion of the boring and collection of the groundwater sample, the direct push tooling will be pulled back to allow formation collapse to install the first soil-gas sampling point. The shallow subsurface geology (less than 30-feet below land surface) in the vicinity of the investigation area is comprised of fine- to coarse-grained unconsolidated glacial deposits. These deposits consist of sand and gravel with little fines. Based on the geology the formation collapse will be homogeneous during installation of the soil-gas sampling points and will not seal off the borehole or create a preferential pathway from the groundwater to the soil-gas sampling points. Appendix C contains selected boring logs in the area of the investigations. The first sampling point will be installed approximately 2 feet above the maximum groundwater table elevation to ensure that the soil-gas sampling point will not be submerged. The soil-gas sampling point will be surrounded by a sand pack to approximately 6 inches above the sampling point. Dry granular bentonite will be placed on top of the sand pack followed by hydrated bentonite to about 2 inches from the base of the next soil-gas screen. Sand will be placed above the bentonite and the next soil-gas sampling point installed at 11 feet bls and surrounded by sand extending 6 inches above the sampling point. Dry granular bentonite will be placed on top of the sand pack followed by hydrated bentonite to approximately 2 inches below the base of the shallowest screen set at 6 feet. Sand will be poured to 6 inches above the final screen and bentonite will be added to approximately 2 feet from the surface to which a well vault will be installed. Approximately 3 feet of bentonite (hydrated on top of dry) will be above the shallowest sample port to ensure no short-circuiting with ambient air. All information regarding the sample port installation will be recorded on the soil-gas point construction diagram (Appendix A).

The soil-gas sampling point will be constructed of a 6-inch stainless steel screened implant connected to Teflon tubing extending to the surface with a clean brass needle valve. After installation, sample points will be allowed to equilibrate for 48-hours prior to sampling (CEPA, 2010).

## 5. Soil-Gas Sampling

A field instrument will be used on-site during sampling to measure temperature, barometric pressure, and relative humidity. Prior to sampling, all points will be checked for leaks by administering helium gas as a tracer with a sealed manifold.

Concentrations of helium will be monitored from Tedlar bags and recorded inside the manifold and from the shallowest screened interval as soil-gas is purged prior to sampling (SOP 37; Appendix A).

A total of three volumes of air (tubing plus screen, sand pack, and dry granular bentonite pore space) will be purged prior to sampling with a flow rate not exceeding 50 milliliters per minute (SOP 36; Appendix A). Soil-gas sample points will be sampled using Method TO-15 for site-specific VOCs. Each soil-gas sample will be collected in a batch certified 250 mL SUMMA®-type canister provided and analyzed by Air Toxics, Folsom, California (Air Toxics). Flow controllers are cleaned, inspected, and set by the analytical laboratory prior to the sampling event. Documentation on completion of batch certification for canisters and cleaning procedures for flow controllers/regulators will be obtained from the Air Toxics and will be included in the summary report. During sampling, the duration of sampling will be monitored to ensure the flow controllers are working properly. All SUMMA ®-type canisters received from Air Toxics will be checked for correct vacuum. The vacuum gauges provided by the analytical laboratory as part of the sample train (i.e., canister and flow controller) are used to record the initial and final vacuums in the air sampling canister. Pre-sampling vacuum in the canister should be between -30 inches of mercury (in Hg) and -25 in Hg. In the event a canister is not within this initial range, it will be rejected and a new canister, flow controller and vacuum will be similarly checked. As a further quality control measure (as outlined in SOP 36; Appendix A) samples will be terminated when the sample duration is finished (approximately 4-minutes) or the canister reaches -5 in Hg, whichever comes first, as a quality control measure. Laboratory methodology and QA/QC information are attached in electronic format in Appendix B. Additional QA/QC samples (duplicates – 1 per 20 samples) will be collected. The draft QAPP with amendments will be followed for this Work Plan (ARCADIS, Inc. 2003).

## 6. Schedule

The field investigation schedule is presented on Figure 10 and outlined below:

- Public meeting, obtain a permit to drill from the City of Moraine (9/27/2010-10/1/2010);
- The field work preparation including subcontractor procurement and contact Ohio Utility Protection Service (OUPS) (10/4/2010-10/15/2010);
- Drilling, groundwater sample collection, and soil-gas sampling point installation (10/18/2010-11/5/2010);
- Soil-gas sampling will be scheduled to capture temporal and seasonal variations and is estimated to take five days to complete; the first round of soil-gas samples from the soil-gas sampling points will be completed the week following the drilling and soil-gas sampling point installation in the fall (11/8/2010-11/12/2010); and
- The second soil-gas sampling will be completed in the spring (3/14/2011-3/18/2011).

During the first soil gas sampling event an internal audit will be conducted to confirm adherence to field procedures outlined in the work plan and SOPs. Based on the first round of data results, immediate action including sub-slab soil-gas and indoor air sampling may be necessary, prior to collecting a second round of soil vapor data. Upon completion of the first round of soil-gas sampling in November 2010, a data package containing the soil-gas analytical results will be presented to the U.S. EPA. A summary report will be prepared and submitted to the U.S. EPA after both sampling events are completed (April 25, 2011).

## 7. Reporting

Upon completion of the first round of soil-gas sampling in November 2010, a data package containing the soil-gas analytical results will be presented to the U.S. EPA. After completion of the second round of soil-gas sampling and receipt of the analytical data, a summary letter report for both sampling events will be completed and submitted to the U.S. EPA. The report will include a summary of all the data collected including quality assurance/quality control samples, data validation report, discussion of results, and as appropriate, recommendations for future work related to soil-gas at the Site. The generic screening criteria provided from US EPA (2002) guidance will be used as part of the overall weight of evidence for evaluating the soil-gas results collected in the right-of-way (Table 4). If soil-gas results are below the generic screening criteria, this will provide another line of evidence that the vapor intrusion pathway is incomplete. Additional considerations will include shallow groundwater data results, the distance to VOCs in groundwater, the presence of utility lines, and the risk assessment results. The report will include a figure of the actual location of all soil-gas sampling points which were installed and a tabular result of all soil-gas samples and groundwater samples collected as part of this investigation. Attachments to the report will include the geotechnical data, laboratory data, and soil boring logs completed as part of this investigation.

**8. References**

ARCADIS, Inc., 2010. Draft Site-Wide Groundwater Monitoring Report for 2009, Motors Liquidation Company, Moraine, Ohio. February 2010.

ARCADIS, Inc., 2008. Draft Corrective Measures Proposal, General Motors Corporation, Moraine, Ohio. August 2008.

ARCADIS Inc., 2007. Corrective Measures Supplemental Groundwater Investigation Report, General Motors Corporation, Moraine, Ohio. February 28, 2007.

ARCADIS Inc., 2003. Draft Quality Assurance Project Plan, General Motors Corporation, Moraine, Ohio. 2003.

ARCADIS Geraghty & Miller, Inc., 2000a. Resource Conservation and Recovery Act Facility Investigation Report - Volume I (Methodologies and Results) Delphi Harrison Thermal Systems, General Motors Corporation, Moraine, Ohio. April 2000.

ARCADIS Geraghty & Miller, Inc., 2000b. Supplemental RFI - Volume I (Methodologies and Results) General Motors Powertrain Group Moraine Engine Plant and General Motors Truck Group Moraine Assembly Plant, Moraine, Ohio. April 2000.

CEPA, 2010: California Environmental Protection Agency. 2010. "Advisory – Active Soil Investigation". March.

[http://www.dtsc.ca.gov/SiteCleanup/upload/SAG\\_Review\\_Drft.pdf](http://www.dtsc.ca.gov/SiteCleanup/upload/SAG_Review_Drft.pdf)

ENVIRON Corporation, 2000a. RCRA Facility Investigation Final Report Volume II (Baseline Risk Assessment), Delphi Harrison Thermal Systems, General Motors Corporation, Moraine, Ohio. April 2000.

ENVIRON Corporation, 2000b. Supplemental Resource Conservation and Recovery Act Facility Investigation Report, Volume II Supplemental Baseline Risk Assessment, General Motors Powertrain Group Moraine Engine Plant and General Motors Truck Group Moraine Assembly Plant, Moraine, Ohio. April 2000.

U.S. Environmental Protection Agency, 2002. OSWER Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils (Subsurface Vapor Intrusion Guidance), EPA530-D-02-004. November 2002.

Table 1. Property Information Details for Neighborhood Located Southwest of Site, Motors Liquidation Company, Moraine, Ohio.

Parcel ID Number	Owner	Addresses	Acres	Basement/ Crawl Space	Year Built	Square Footage	Number of Proposed Sub- Slab Points	No. of Rooms	No. of Floors	Land Use	
J441210060001		3005 Old Sellars Road	0.1212	Crawl	1942	780	N/A <sup>1</sup>	5	1	Residential	
J441210060002			0.1136								
J441210060003		3009 Old Sellars Road	0.1136	Full	1942	989	1	5	1	Residential	
J441210060004		3013 Old Sellars Road	0.1136	None	1942	1728	2	8	2	Residential	
J441210060005		3017 Old Sellars Road	0.1136	Crawl	1967	1008	N/A <sup>1</sup>	5	1	Residential	
J441210060006		3021 Old Sellars Road		0.1136	None	1937	1200	2	5	2	Residential
J441210060007				0.079							
J441210060024		3022 Lakehurst	0.1061	Full	1957	780	1	5	1	Residential	
J441210060025											
J441210060026		3020 Lakehurst	0.1061	Part	1943	720	1	4	1	Residential	
J441210060027		3018 Lakehurst	0.1061	Part	1942	945	1	4	1	Residential	
J441210060028		3016 Lakehurst	0.1061	Crawl	1967	1404	N/A <sup>1</sup>	7	1	Residential	
J441210060029		3010 Lakehurst	0.1061	Crawl	1968	1008	N/A <sup>1</sup>	5	1	Residential	
J441210060030		3000 Lakehurst		0.1061	Full	1942	897	1	5	1	Residential
J441210060031				0.0985							
J441210060032		2920 Lakehurst	0.146	Part	1940	1296	2	7	1.5	Residential	
J441210060033		2916 Lakehurst	0.1229	Part	1944	528	1	4	1	Residential	
J441210060034		2912 Lakehurst	0.1212	Full	1942	997	1	4	1	Residential	
J441210060035		2908 Lakehurst	0.1858	Crawl	1925	816	N/A <sup>1</sup>	4	1	Residential	
J441210060042		2909 Sellars Road	0.2314	None	1937	1095	2	5	1	Residential	
J441210060044		2917 Old Sellars	0.1157	Full	1939	840	1	5	1	Residential	
J441210060045		2921 Old Sellars		0.1157	Full	1933	1072	2	6	1	Residential
J441210060046				0.1074							
J441210060041		2901 Dryden Rd./Main		0.1157	None	Various	960	2	N/A	1	Commercial
J441210060056				0.2925			9000		N/A	1	
J441210060057				0.352			3120		N/A	1	
J441210060058				0.186			3024		N/A	1	
J441210070001		2924 Telhurst		0.1377	Part	1940	1080	1	5	1	Residential
J441210070002				0.124							
J441210070003		2916 Telhurst	0.124	Full	1944	874	1	6	1	Residential	
J441210070004		2912 Telhurst	0.124	Full	1944	1159	2	6	1	Residential	
J441210070005		2908 Telhurst		0.124	Crawl	1935	1210	N/A <sup>1</sup>	5	1	Residential
J441210070006				0.0634							
J441210070007	2900 Telhurst		0.1295	Part	1910	1174	2	6	1	Residential	
J441210070021			0.0909								
J441210070008	2925 Lakehurst	0.1405	Crawl	1945	1180	N/A <sup>1</sup>	6	1	Residential		
J441210070009	2921 Lakehurst	0.1157	None	1950	720	1	4	1	Residential		

Table 1. Property Information Details for Neighborhood Located Southwest of Site, Motors Liquidation Company, Moraine, Ohio.

Parcel ID Number	Owner	Addresses	Acres	Basement/ Crawl Space	Year Built	Square Footage	Number of Proposed Sub- Slab Points	No. of Rooms	No. of Floors	Land Use
J441210070010		2917 Lakehurst	0.1157	None	1940	720	1	5	1	Residential
J441210070011		2913 Lakehurst	0.1157	Part	1942	1022	1	5	1	Residential
J441210070012		2905 Lakehurst	0.1157	Part	1939	1208	2	6	1	Residential
J441210070013		2901 Lakehurst	0.146	Crawl	1942	1085	N/A <sup>1</sup>	6	1	Residential
J441210070014		3413 Dryden Road	0.1056	Part	1940	1078	1	5	1	Residential
J441210070015		3409 Dryden	0.1056	Crawl	1940	720	N/A <sup>1</sup>	5	1	Residential
J441210070016		3401 Dryden	0.1056	Crawl	1935	1924	N/A <sup>1</sup>	6	2	Residential
J441210070017		2925 Telhurst	0.1736	None	1971	909	1	5	1	Residential
J441210070018		2921 Telhurst	0.124	None	1971	962	1	5	1	Residential
J441210070019		2913 Telhurst	0.2479	Part	1971	1272	2	6	1	Residential
J441210070020		2909 Telhurst	0.124	Crawl	1948	1008	N/A <sup>1</sup>	6	1	Residential
J441210080001		2905 Telhurst	0.124	Crawl	1948	700	N/A <sup>1</sup>	4	1	Residential
J441210080002		2901 Telhurst	0.1295	None	1953	912	1	5	1	Residential
J441210080003		3391 Dryden Road	0.1148	None	1944	806	1	5	1	Residential
J441210080004		2900 Hoylake	0.1488	Full	1950	1092	2	5	1	Residential
J441210080005		2904 Hoylake	0.1157	Full	1950	956	1	5	1	Residential
J441210080006		2908 Hoylake	0.1157	Crawl	1954	800	N/A <sup>1</sup>	4	1	Residential
J441210080007		2912 Hoylake	0.1157	Crawl	1950	576	N/A <sup>1</sup>	4	1	Residential
J441210080008		2916 Hoylake	0.1157	None	1950	1690	2	6	1	Residential
J441210080009		2924 Hoylake	0.1157	Part	1950	996	1	6	1	Residential
J441210080010		2928 Hoylake	0.135	Part	1956	1152	2	6	1.5	Residential
J441210080011		3001 Telhurst	0.1488	Crawl	1949	876	N/A <sup>1</sup>	6	1	Residential
J441210080012		3005 Telhurst	0.1157	Part	1948	982	1	5	1	Residential
J441210080013		3009 Telhurst	0.1157	Part	1957	760	1	5	1	Residential
J441210080014		3013 Telhurst	0.1157	Crawl	1947	870	N/A <sup>1</sup>	4	1	Residential
J441210080015		3015 Telhurst	0.1157	Crawl	1947	624	N/A <sup>1</sup>	4	1	Residential
J441210080016		3020 Hoylake	0.124	Crawl	1973	912	N/A <sup>1</sup>	5	1	Residential
J441210080017		3018 Hoylake	0.124	Crawl	1973	912	N/A <sup>1</sup>	5	1	Residential

Table 1. Property Information Details for Neighborhood Located Southwest of Site, Motors Liquidation Company, Moraine, Ohio.

Parcel ID Number	Owner	Addresses	Acres	Basement/ Crawl Space	Year Built	Square Footage	Number of Proposed Sub- Slab Points	No. of Rooms	No. of Floors	Land Use
J441210080051		3016 Hoylake	0.124	Full	1947	672	1	4	1	Residential
J441210080052		3012 Hoylake	0.1157	Crawl	1947	640	N/A <sup>1</sup>	4	1	Residential
J441210080053		3008 Hoylake	0.1157	Crawl	1947	1016	N/A <sup>1</sup>	6	1	Residential
J441210080054			0.124							
J441210080055		3000 Hoylake	0.1185	Crawl	1953	528	N/A <sup>1</sup>	4	1	Residential
J441210090014		3012 Telhurst	0.2287	Part	1963	1380	2	6	1	Residential
J441210090015		3006 Telhurst	0.2287	Part	1963	1380	2	6	1	Residential
J441210090001		3001 Lakehurst	0.2369	None	Not available	1755	2	1	1	Commercial
J441210090002			0.4573							
J441210090016			0.2204							
J441282230001			3901 Dryden Road							

ID - Identification.

Parcel IDs, addresses, owners, and acreage found on Montgomery County Auditors web site ([www.mcauditor.org](http://www.mcauditor.org)).

No. - Number

Crawl - Crawl space underneath structure.

Part - Partial basement underneath structure.

Full - Full basement underneath structure.

None - No sub-grade construction underneath structure.

N/A - Not applicable.

1 - Sub-slab points are not installed in a crawl space.

# ARCADIS

Table 2. Recent Groundwater Sampling Data and Vertical Aquifer Profiling Data from Select Monitoring Wells, Motors Liquidation Company, Moraine, Ohio.

Location	In Neighborhood Southwest of Site									On Eastern Boundary of Site					
	Well ID	GM-15	GM-16	GM-47				GM-50	GM-63	GM-64	GM-25	GM-53	GM-54	GM-53/54	
Date	11/13/09	11/13/09	2/23/2006	2/24/2006	11/13/09	2/24/2006	11/13/09	4/29/09	4/29/09	9/22/99	9/14/06	11/13/09	7/12/06	7/12/06	
Well Screen <sup>1</sup>	90-100	48-58	25-30 ft	40-45 ft	49.4-59.4	55-60 ft	29.7-39.7	30-40	50-60	48-58	23-33	70-80	95-100 <sup>2</sup>	110-115 <sup>2</sup>	
Upper/Lower Aquifer	Lower	Upper	Upper	Upper	Upper	Upper	Upper	Upper	Upper	Upper	Upper	Lower	Lower	Lower	
<b>Volatile Organic Compounds (VOCs)</b>	<b>Units</b>														
1,1,1-Trichloroethane	µg/L	< 1.0 U	1.4 J	3.1	3.2	1 J	2.7	1.8 J	2.1 J	0.38 J	< 1.0 U	< 1.0 U	< 5.0 U	< 1 U	< 1 U
1,1-Dichloroethane	µg/L	1.2	1.6 J	1.6	1.6	2.4 J	1.9	1.3 J	1.6 J	3.7	< 1.0 U	< 1.0 U	< 5.0 U	1.1	< 1 U
1,1-Dichloroethene	µg/L	< 1.0 U	< 3.3 U	< 1 U	< 1 U	< 2.5 U	< 1 U	< 5.0 U	< 5.0 U	< 1.0 U	< 1.0 U	< 1.0 U	< 5.0 U	0.35 J	< 1 U
Benzene	µg/L	< 1.0 U	< 3.3 U	< 1 U	< 1 U	< 2.5 U	< 1 U	< 5.0 U	< 5.0 U	< 1.0 U	< 1.0 U	< 1.0 U	< 5.0 U	< 1 U	< 1 U
cis-1,2-Dichloroethene	µg/L	1	12	4.6	5.7	61	9.8	11	6.2	11	< 1.0 U	< 1.0 U	< 5.0 U	6.2	3.9
Ethylbenzene	µg/L	< 1.0 U	< 3.3 U	< 1 U	< 1 U	< 2.5 U	< 1 U	< 5.0 U	< 5.0 U	< 1.0 U	< 1.0 U	< 1.0 U	< 5.0 U	< 1 U	< 1 U
Tetrachloroethene	µg/L	< 1.0 U	110	135	143	61	69	110	110	12	< 1.0 U	< 1.0 U	120	1	0.95 J
Toluene	µg/L	< 1.0 U	< 3.3 U	< 1 U	< 1 U	< 2.5 U	< 1 U	< 5.0 U	< 5.0 U	< 1.0 U	< 1.0 U	< 1.0 U	< 5.0 U	< 1 U	< 1 U
trans-1,2-Dichloroethene	µg/L	< 1.0 U	1.5 J	1.4	1.4	2.8	2.1	< 5.0 U	< 5.0 U	2.2	< 1.0 U	< 1.0 U	< 5.0 U	0.37 J	0.55 J
Trichloroethene	µg/L	6.5	74	110	112	29	49.8	120	120	8.6	< 1.0 U	< 1.0 U	2.9 J	0.42 J	0.33 J
Vinyl chloride	µg/L	< 1.0 U	< 3.3 U	< 1 U	< 1 U	0.66 J	< 1 U	< 5.0 U	< 5.0 U	2.5	< 1.0 U	< 1.0 U	< 5.0 U	0.49 J	0.23 J
Xylene (total)	µg/L	< 2.0 U	< 6.7 U	0.73 J	0.69 J	< 5.0 U	< 2 U	< 10 U	< 10 U	< 2.0 U	< 1.0 U	< 2.0 U	< 10 U	< 2 U	< 2 U
<b>Total VOCs</b>	<b>µg/L</b>	<b>8.7</b>	<b>200.5</b>	<b>256.43</b>	<b>267.59</b>	<b>157.86</b>	<b>135.3</b>	<b>244.1</b>	<b>239.9</b>	<b>40.38</b>	<b>0</b>	<b>0</b>	<b>122.9</b>	<b>9.93</b>	<b>5.96</b>

µg/L - Micrograms per Liter.

J - Value is estimated.

< - Constituent not detected above laboratory reporting limit shown.

U - Constituent not detected.

1 - Well Screen feet below land surface.

2 - Sampling depth of vertical aquifer profiling groundwater sample.

Table 2. Recent Groundwater Sampling Data and Vertical Aquifer Profiling Data from Select Monitoring Wells, Motors Liquidation Company, Moraine, Ohio.

	Location	On Eastern Boundary of Site								East of Site							
	Well ID	GM-58				GM-70				GM-77S	GM-77D	GM-77	GM-84				
	Date Well Screen <sup>1</sup> Upper/Lower Aquifer	8/22/06 25-30 <sup>2</sup> Upper	9/26/08 72-82 Lower	8/22/06 95-100 <sup>2</sup> Lower	8/22/06 110-115 <sup>2</sup> Lower	4/5/07 40-45 <sup>2</sup> Upper	4/5/07 93-98 <sup>2</sup> Lower	4/5/07 112-117 <sup>2</sup> Lower	5/3/07 72-82 Lower	9/27/07 33-43 Upper	9/27/07 75-85 Lower	9/7/07 90-95 <sup>2</sup> Lower	2/1/08 32-37 ft Upper	2/4/08 52-57 ft Upper	2/4/08 68-73 ft Upper	2/5/08 95-100 ft Lower	2/5/08 115-120 ft Lower
<b>Volatile Organic Compounds (VOCs)</b>	<b>Units</b>																
1,1,1-Trichloroethane	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	0.21 J	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
1,1-Dichloroethane	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
1,1-Dichloroethene	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
Benzene	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
cis-1,2-Dichloroethene	µg/L	< 1 U	< 2.5 U	2.4	3.5	< 1 U	1	2.3	< 1.0 U	< 1.0 U	< 1.4 U	0.33 J	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
Ethylbenzene	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
Tetrachloroethene	µg/L	< 1 U	80	0.69 J	0.26 J	4.5	2	1.7	11	< 1.0 U	45	2.6 J	1.4	2.1	2.4	< 1 U	< 1 U
Toluene	µg/L	0.38 J	< 2.5 U	0.27 J	0.34 J	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	0.42 J	< 1 U	0.51 J	0.36 J	0.49 J
trans-1,2-Dichloroethene	µg/L	< 1 U	< 2.5 U	< 1 U	0.22 J	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
Trichloroethene	µg/L	< 1 U	< 2.5 U	0.91 J	< 1 U	0.56 J	7.1	1.2	0.32 J	< 1.0 U	< 1.4 U	< 1 UJ	0.8 J	7	7.1	5.3	3
Vinyl chloride	µg/L	< 1 U	< 2.5 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U	< 1.0 U	< 1.0 U	< 1.4 U	< 1 UJ	< 1 U	< 1 U	< 1 U	< 1 U	< 1 U
Xylene (total)	µg/L	< 2 U	< 5.0 U	< 2 U	< 2 U	< 2 U	< 2 U	< 2 U	< 2.0 U	< 2.0 U	< 2.9 U	< 2 UJ	< 2 U	< 2 U	< 2 U	< 2 U	< 2 U
<b>Total VOCs</b>	<b>µg/L</b>	<b>0.38</b>	<b>80</b>	<b>4.27</b>	<b>4.32</b>	<b>5.48</b>	<b>10.1</b>	<b>5.2</b>	<b>11.32</b>	<b>0</b>	<b>45</b>	<b>2.93</b>	<b>2.62</b>	<b>9.1</b>	<b>10.01</b>	<b>5.66</b>	<b>3.49</b>

µg/L - Micrograms per Liter.

J - Value is estimated.

< - Constituent not detected above laboratory reporting limit shown.

U - Constituent not detected.

1 - Well Screen feet below land surface.

2 - Sampling depth of vertical aquifer profiling groundwater sample.

# ARCADIS

**Table 3. Historic Groundwater Fluctuations, Motors Liquidation Company, Moraine, Ohio.**

Well	Surface Elevation	2/27/2006	6/21/2006	9/12/2006	4/30/2007	10/24/2007	3/5/2008	4/24/2008
		2/28/2006	6/22/2006	9/13/2006	5/1/2007	10/25/2007	3/6/2008	4/25/2008
<i>Southwest Neighborhood</i>								
GM-16	725.30*	704.52	706.00	705.05	706.91	704.20	708.07	708.38
GM-47	727.03	704.80	706.30	705.34	708.43	704.53	708.10	708.62
GM-50	727.03	NI	706.24	705.26	708.38	704.47	708.06	708.57
GM-63	726.21	NI	NI	705.61	708.22	704.84	708.15	708.93
GM-64	726.38	NI	NI	705.61	708.71	704.83	708.14	708.91
<i>East Area</i>								
GM-53	730.53	NI	NI	705.64	710.46	706.16	708.43	710.93
GM-71	737.19	NI	NI	NI	711.39	706.92	709.05	711.51
GM-72	737.05	NI	NI	NI	711.07	706.97	709.05	711.51
GM-75S	738.26	NI	NI	NI	NI	706.90	709.06	711.45

All elevations in feet above mean sea level

NI - not installed

\* - approximate

# ARCADIS

**Table 3. Historic Groundwater Fluctuations, Motors Liquidation Company, Moraine, Ohio.**

<b>Well</b>	<b>Surface</b>	<b>11/17/2008</b>	<b>11/9/2009</b>	<b>Maximum</b>	<b>Minimum</b>	<b>Mean</b>
<b>Upper Aquifer Wells</b>	<b>Elevation</b>	<b>11/18/2008</b>	<b>11/10/2009</b>	<b>Groundwater</b>	<b>Groundwater</b>	<b>Groundwater</b>
				<b>Elevation</b>	<b>Elevation</b>	<b>Elevation</b>
<i>Southwest Neighborhood</i>						
GM-16	725.30*	704.45	704.45	708.38	704.20	705.78
GM-47	727.03	704.77	704.78	708.62	704.53	706.19
GM-50	727.03	704.73	704.73	708.57	704.47	706.30
GM-63	726.21	705.07	705.14	708.93	704.84	706.57
GM-64	726.38	705.07	705.09	708.91	704.83	706.62
<i>East Area</i>						
GM-53	730.53	706.60	706.64	710.93	705.64	707.84
GM-71	737.19	707.37	707.52	711.51	706.92	708.96
GM-72	737.05	707.38	707.53	711.51	706.97	708.92
GM-75S	738.26	707.34	707.49	711.45	706.90	708.45

All elevations in feet above mean sea level

NI - not installed

\* - approximate

## ARCADIS

**Table 4. Generic Screening Levels for Soil-Gas, Motors Liquidation Company, Moraine, Ohio.**

<b>Chemical Constituent</b>	<b>Long -Term Screening Level Soil-Gas at Water Table (ug/m<sup>3</sup>) or [ppbv]<sup>a,b</sup></b>
Benzene	310 [98 ppbv]
Ethylbenzene	2200 [510 ppbv]
Toluene	40000 [11000 ppbv]
Xylene(s)	700000 [160000 ppbv]
1,1-Dichloroethane	50000 [12000 ppbv]
1,1-Dichloroethene	20000 [5000 ppbv]
Cis-1,2-Dichloroethene	3500 [880 ppbv]
Trans-1,2-Dichloroethene	7000 [1800 ppbv]
Tetrachloroethene	410 [60 ppbv]
1,1,1-Trichloroethane	220000 [40000 ppbv]
Trichloroethene	1200 [220 ppbv]
Vinyl chloride	280 [110 ppbv]

a - These values are based on the "Generic Screening Levels" from Table 2b of the *Draft Guidance for Evaluating the Vapor Intrusion to Indoor Air Pathway from Groundwater and Soils* (U.S. EPA 2002). For the constituents Tetrachloroethene and Trichloroethene, the Indoor Air screening levels were revised by applying EPA's current practice of employing the California EPA Inhalation Unit Risk Factors as the provisional inhalation cancer potency factors.

For Tetrachloroethene - the California EPA IUR is  $5.9E-06 \text{ (ug/m}^3\text{)}^{-1}$

For Trichloroethene - the California EPA IUR is  $2.0E-06 \text{ (ug/m}^3\text{)}^{-1}$

b - The attenuation factor for Indoor Air to Soil-Gas at the Water Table is 0.01.

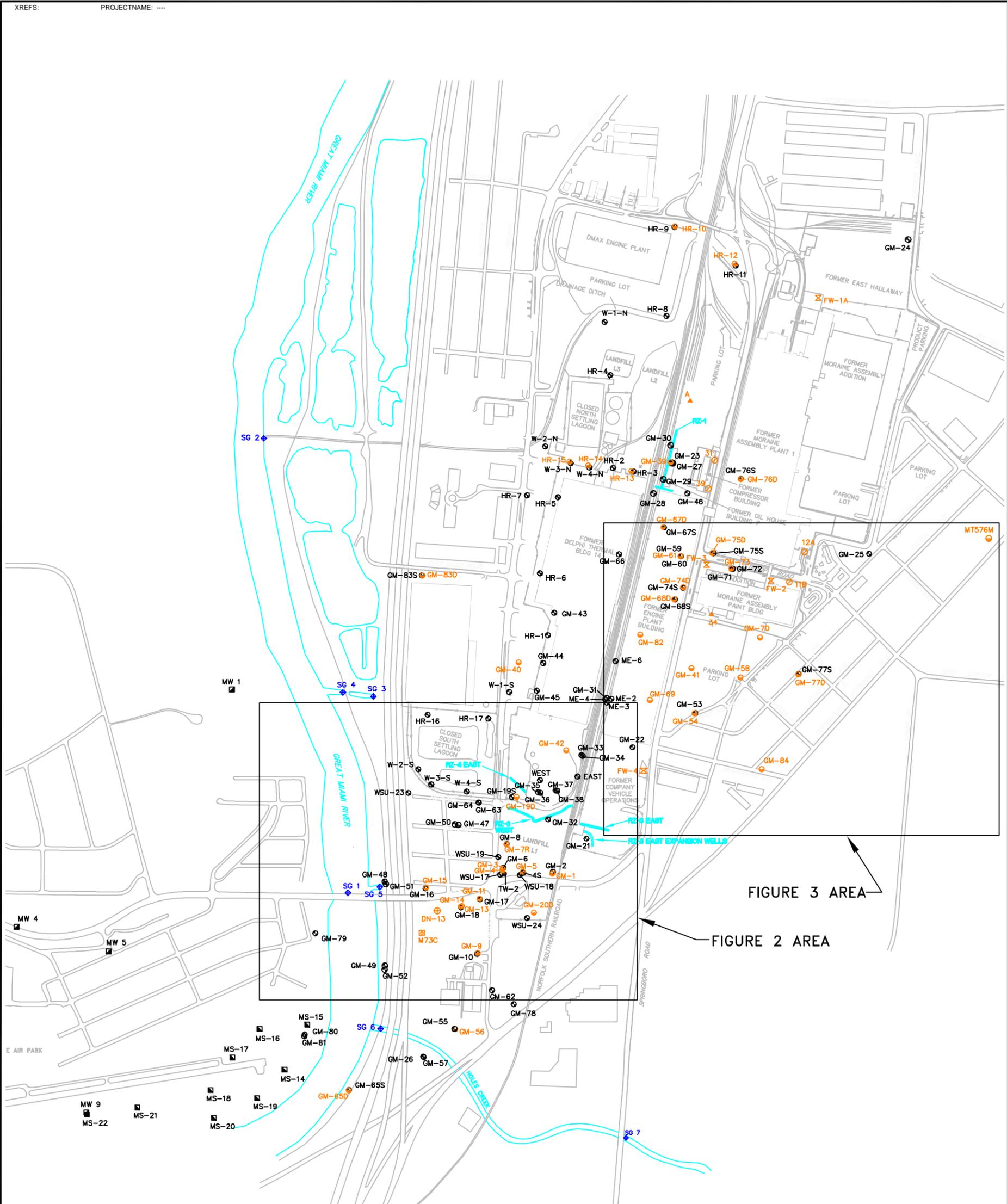


FIGURE 3 AREA

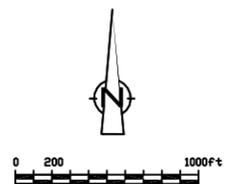
FIGURE 2 AREA

**LEGEND**

- MONITORING WELL (UPPER AQUIFER)
- RECOVERY WELL (TW-2)
- MONITORING WELL (LOWER AQUIFER)
- PIEZOMETER
- CARBON SOURCE INTRODUCTION WELLS, REACTIVE ZONES (RZ-1, RZ-3, AND RZ-4)
- FIRE WELL
- PRODUCTION WELL CONVERTED TO MONITORING WELL (34, A)
- INACTIVE PRODUCTION WELL (31, 39, 11B, 12A)
- MONTGOMERY COUNTY WELL (USED BY MLC FOR PUMP TO WASTE PROGRAM)
- MONTGOMERY COUNTY WELL (INACTIVE MIAMI SHORES WELL FIELD - DAYTON PRIMARY PUBLIC SUPPLY BACKUP)
- STREAM GAUGE
- RIVER LEVEE
- CITY OF MORAINES MONITORING WELL
- FORMER BUILDING FOOTPRINT
- SURFACE WATER FEATURE

**NOTES:**

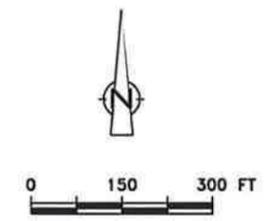
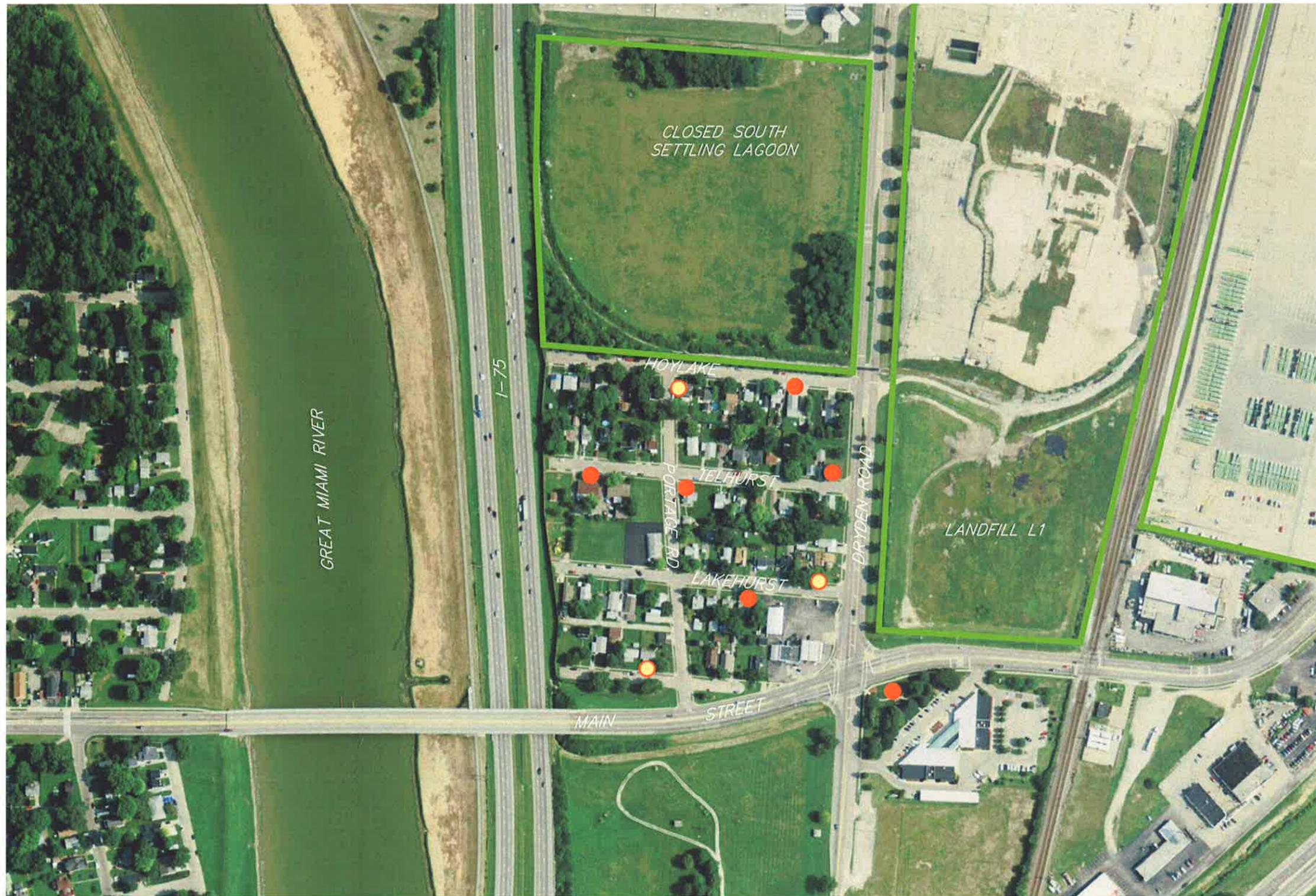
1. ORANGE INDICATES LOWER AQUIFER WELLS.
2. BLACK INDICATES UPPER AQUIFER WELLS.



MOTORS LIQUIDATION COMPANY  
 MORAINES, OHIO  
 OH000294.0016

**SITE LAYOUT**

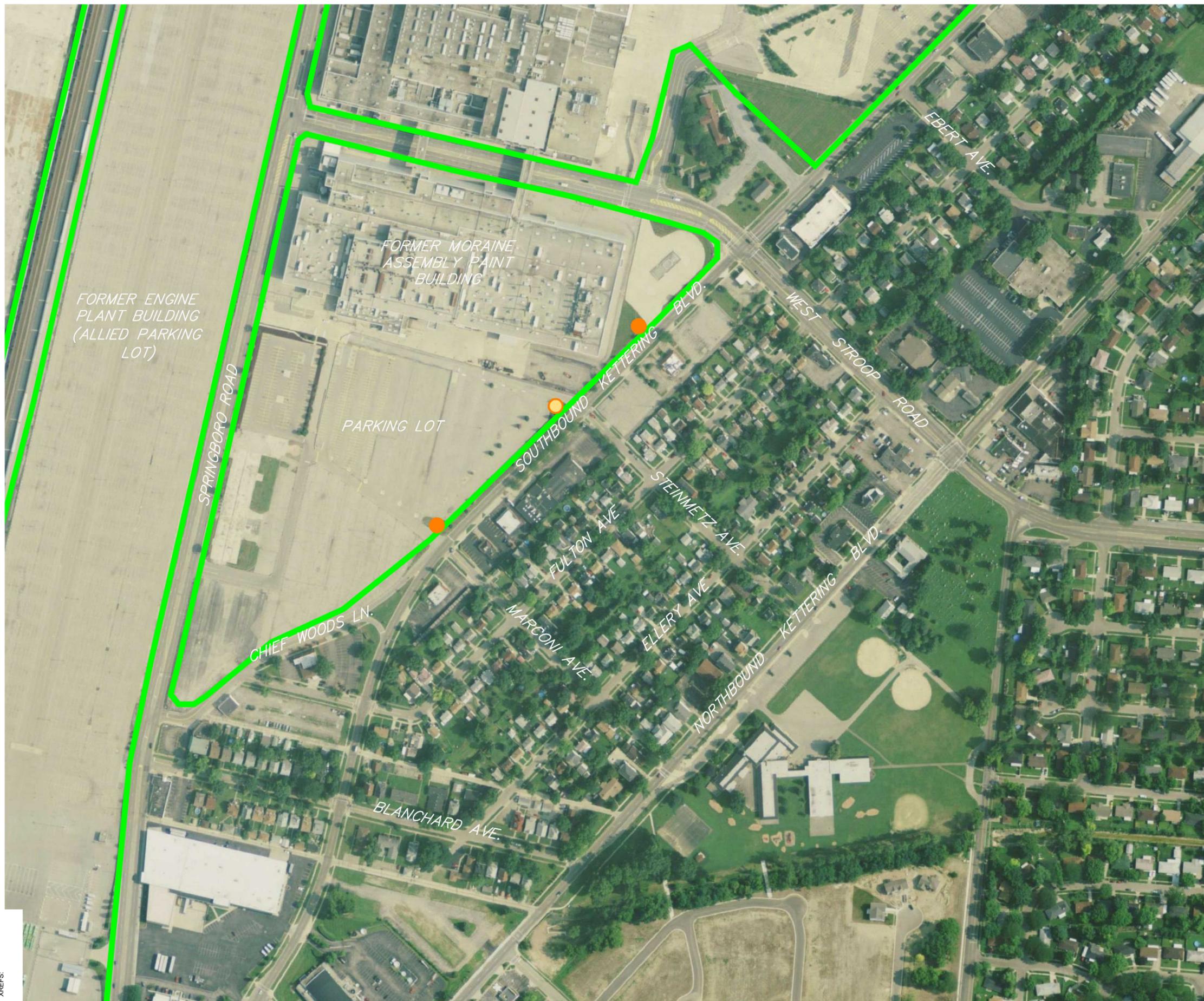




- LEGEND**
- PROPOSED SOIL-GAS SAMPLING POINT
  - PROPOSED SOIL-GAS SAMPLING POINT AND SHELBY TUBE SAMPLING LOCATION
  - MLC PROPERTY BOUNDARY

MOTORS LIQUIDATION COMPANY MORAIN, OHIO OH000294.0016	
<b>AERIAL PHOTOGRAPH VIEW OF                  NEIGHBORHOOD SOUTHWEST                  OF SITE</b>	
	FIGURE <b>2</b>

CITY:(COLUMBUS) DIV:(GROUP:USER1) DB:(R. SMITH) LD:(Op) PIC:(Op) PM:(N. GILLOTTI) TM:(OPT) LVR:(OP)ON="OFF=REF" PLOTSTYLETABLE: ACAD.CTB PLOTTED: 6/3/2010 3:13 PM BY: SMITH, BOB  
G:\ENVCAD\Columbus-ORHACT\01000294-MOTORS LIQUIDATION COMPANY\2010\010294-10-22.dwg LAYOUT: NEIGHBORHOOD EAST OF SITE SAVED: 6/2/2010 3:07 PM ACADVER: 17.05 (LMS TECH) PAGESETUP: ---- PLOTSTYLETABLE: ACAD.CTB PLOTTED: 6/3/2010 3:13 PM BY: SMITH, BOB  
XREFS:



- LEGEND**
- PROPOSED SOIL-GAS SAMPLING POINT
  - PROPOSED SOIL-GAS SAMPLING POINT AND SHELBY TUBE SAMPLING LOCATION
  - MLC PROPERTY BOUNDARY

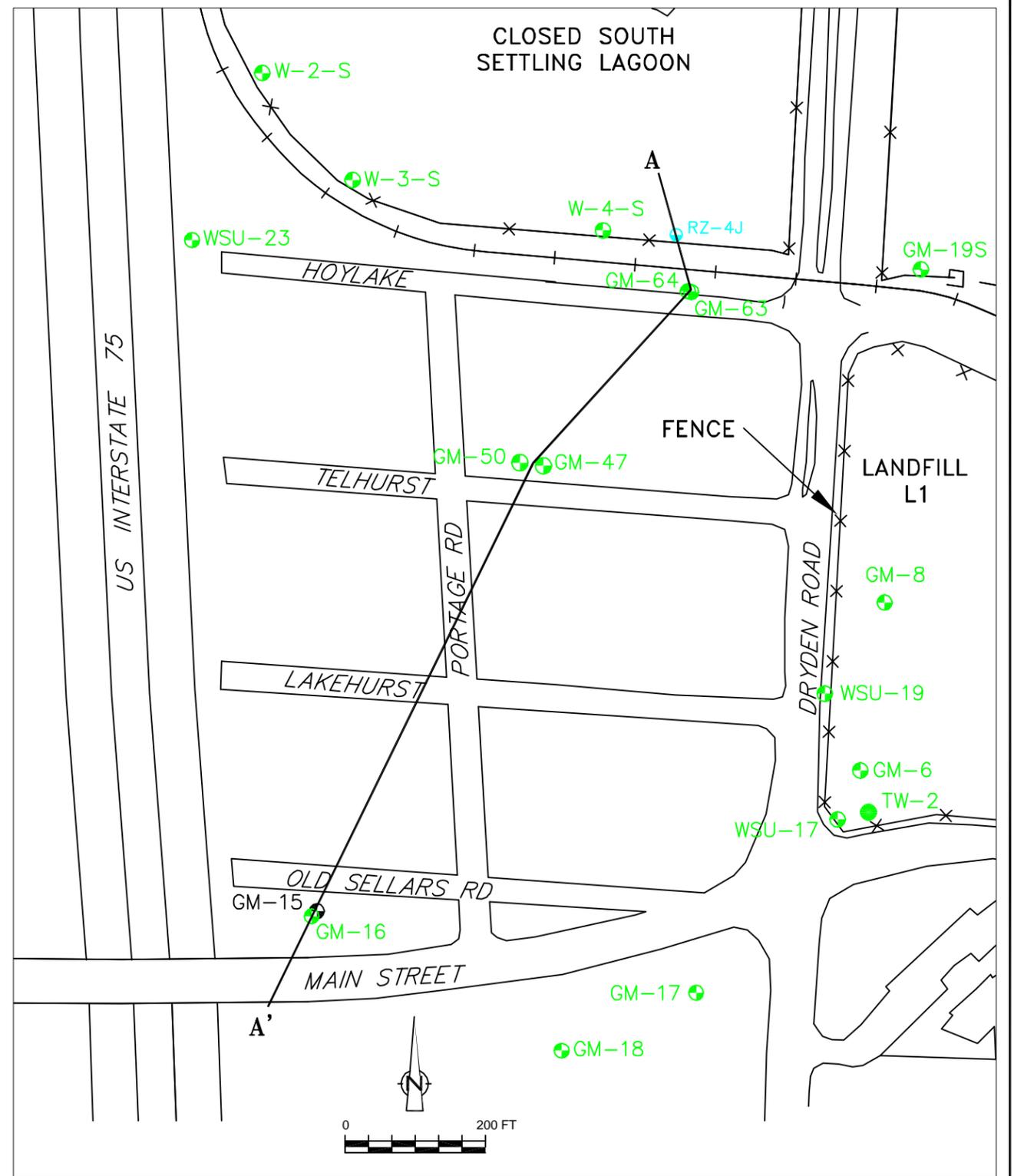
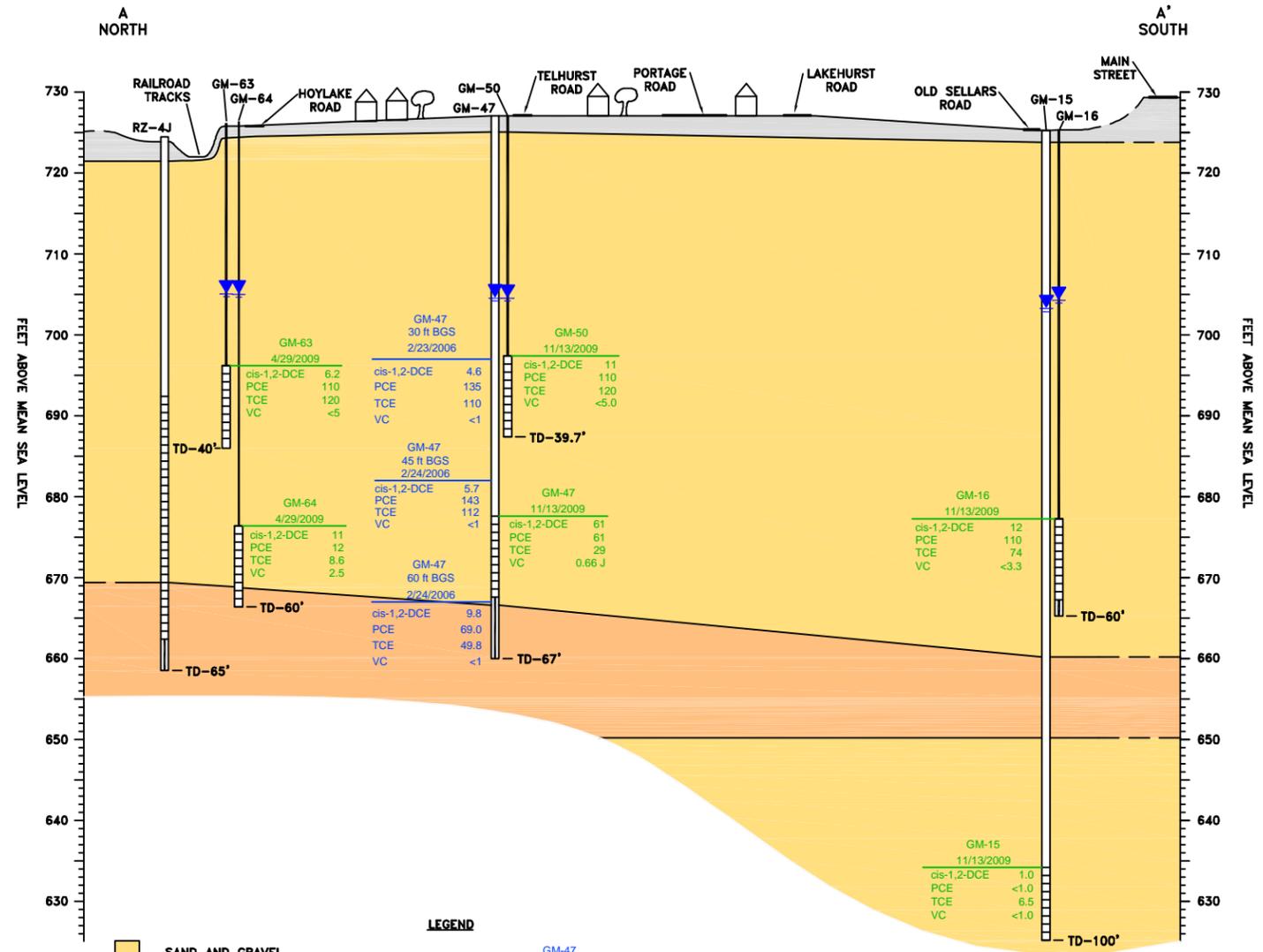
MOTORS LIQUIDATION COMPANY  
MORaine, OHIO  
OH000294.0016

**AERIAL PHOTOGRAPH VIEW  
EAST OF SITE**



FIGURE  
**3**

CITY: COLUMBUS DIV: GROUP: SER: 1 DB: (R. SMITH) LD: (OP) PIC: (OP) PM: (N. GILLOTT) TM: (OPT) LYR: (OPTION) OFF: REF  
 G:\ENV\GAD\columbus\CHACTON\02294\MOTORS LIQUIDATION COMPANY\2010\GSS294\10-01.dwg LAYOUT: CS-A-A' SAVED: 3/6/2010 2:56 PM ACADVER: 17.0S (LIMS TECH) PAGESETUP: --- PLOTSTYLETABLE: ACAD.CTB PLOTTED: 3/11/2010 11:38 AM BY: SMITH, BOB  
 XREFS: IMAGES: cross section a-a.jpg cross section b-b.jpg



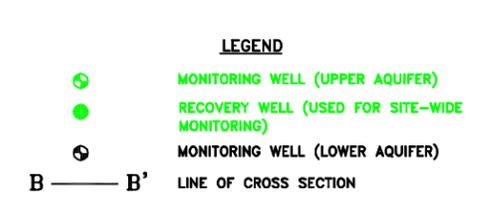
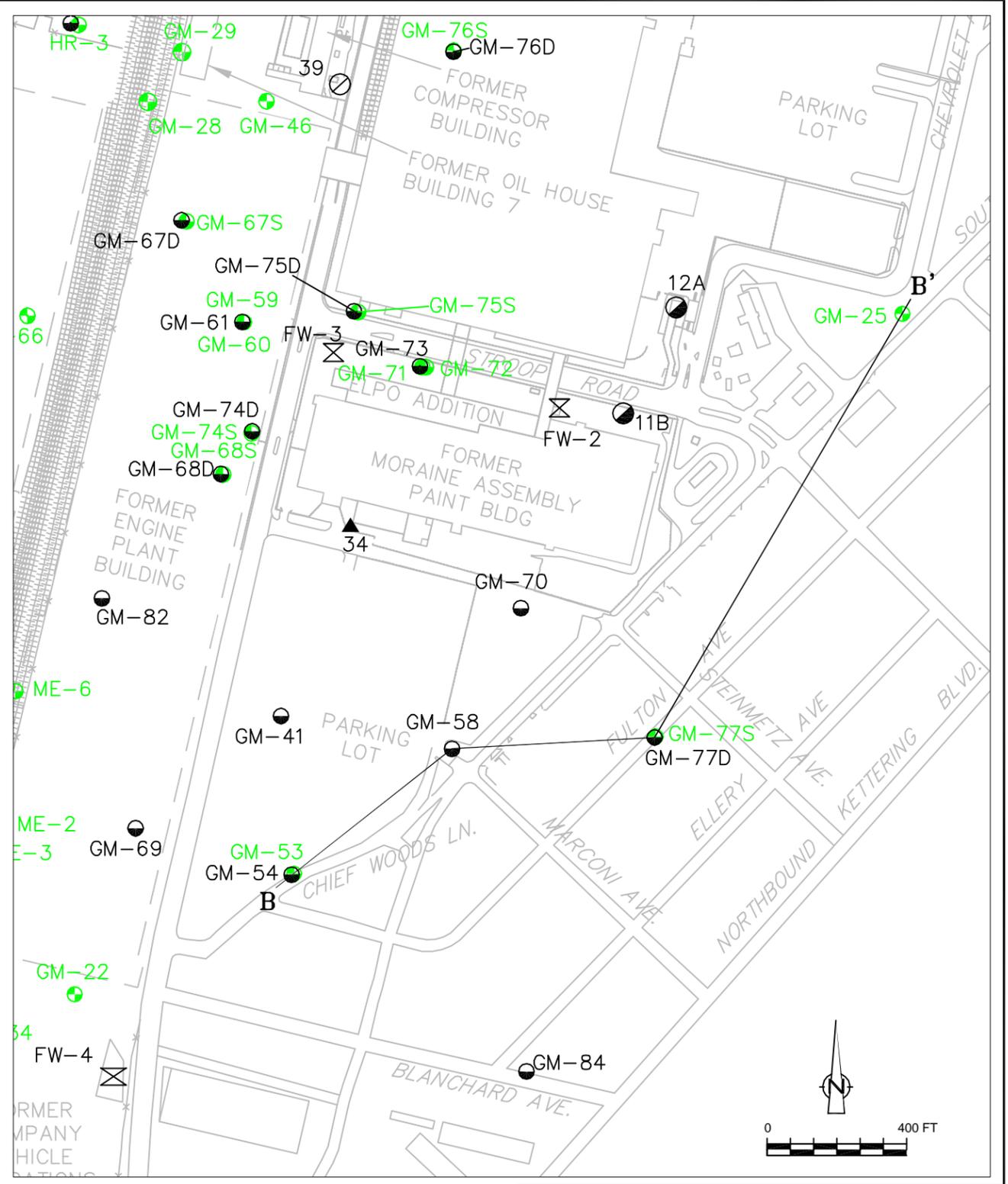
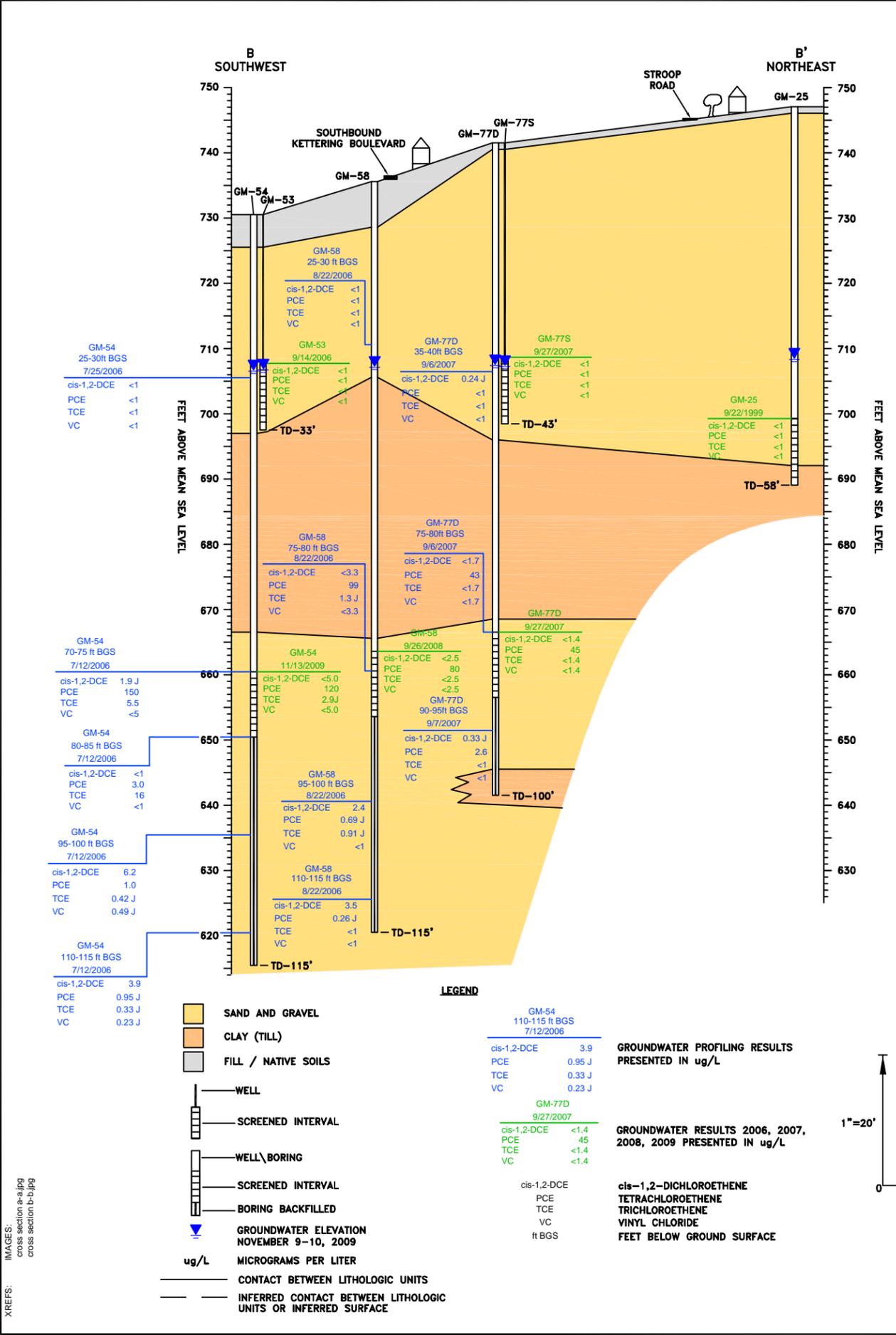
MOTORS LIQUIDATION COMPANY  
MORAIN, OHIO  
OH000294.0016

**CROSS SECTION A-A'**

**ARCADIS**

FIGURE  
**4**

CITY: COLUMBUS DIV: GROUP: SER1 DB: (R. SMITH) LD: (OP) PM: (N. GILLOTT) TM: (OPT) LYR: (OPTION) OFF: REF G: (ENV) CAD: (G) CHACTOR: (H) 00294: (MOTORS LIQUIDATION COMPANY) 2010: (SS) 294: (10:01) .dwg LAYOUT: CS: B-B SAVED: 3/6/2010 2:56 PM ACADVER: 17.0S (LMS TECH) PAGESETUP: PLOTSTYLETABLE: ACAD.CTB PLOTTED: 3/11/2010 11:38 AM BY: SMITH, BOB

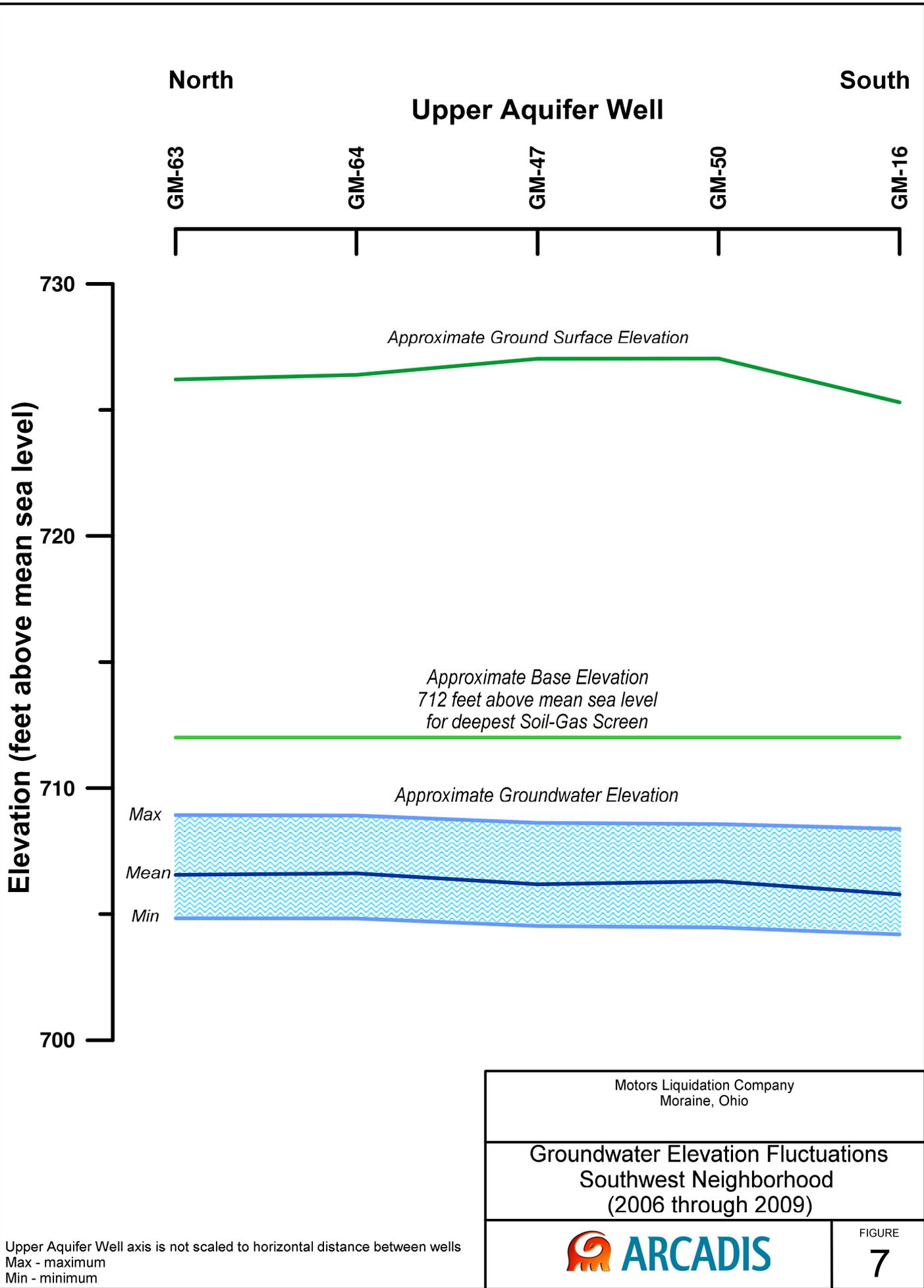


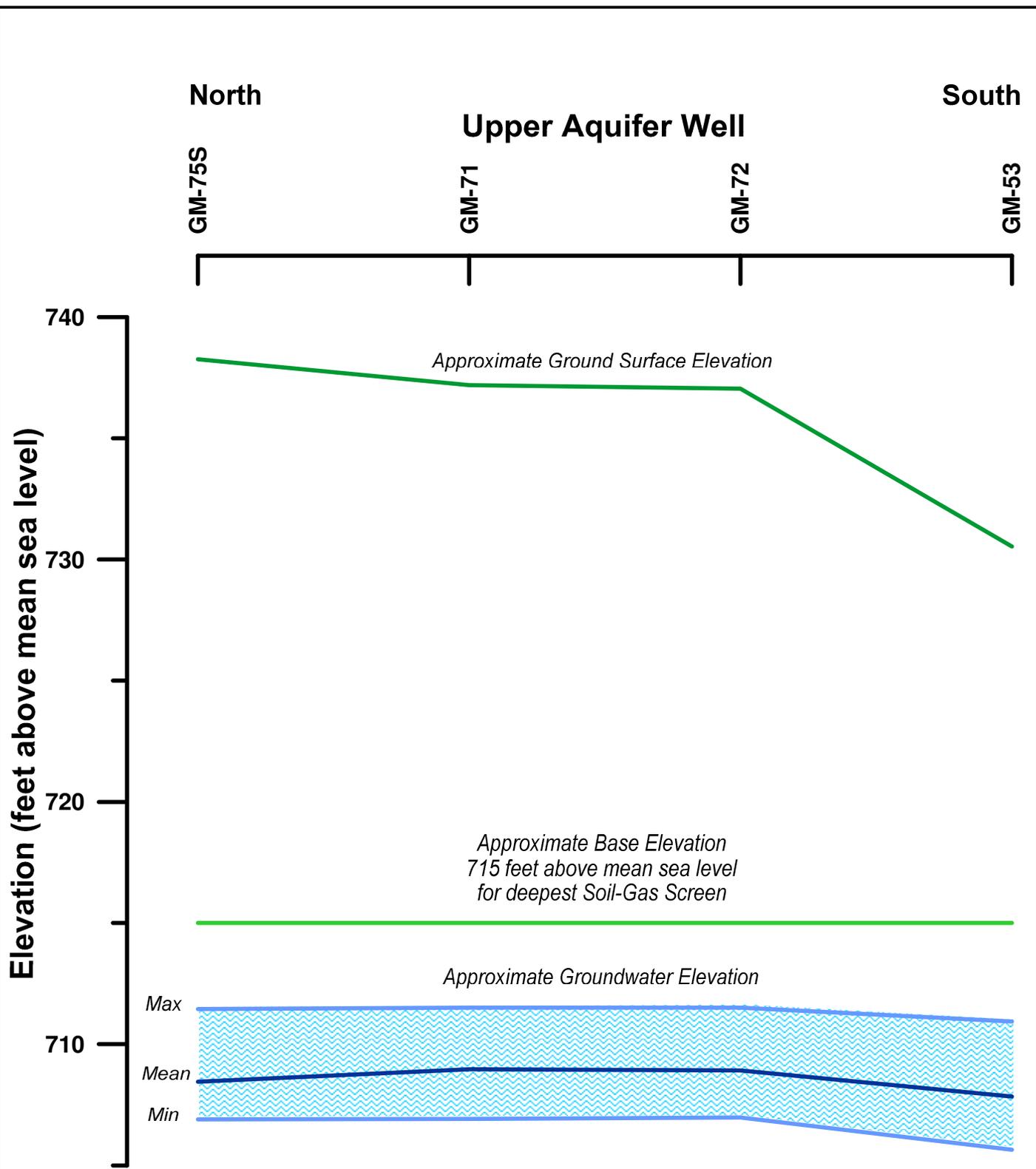
MOTORS LIQUIDATION COMPANY  
MORAINE, OHIO  
OH000294.0016

**CROSS SECTION B-B'**

FIGURE  
**5**







Motors Liquidation Company  
Moraine, Ohio

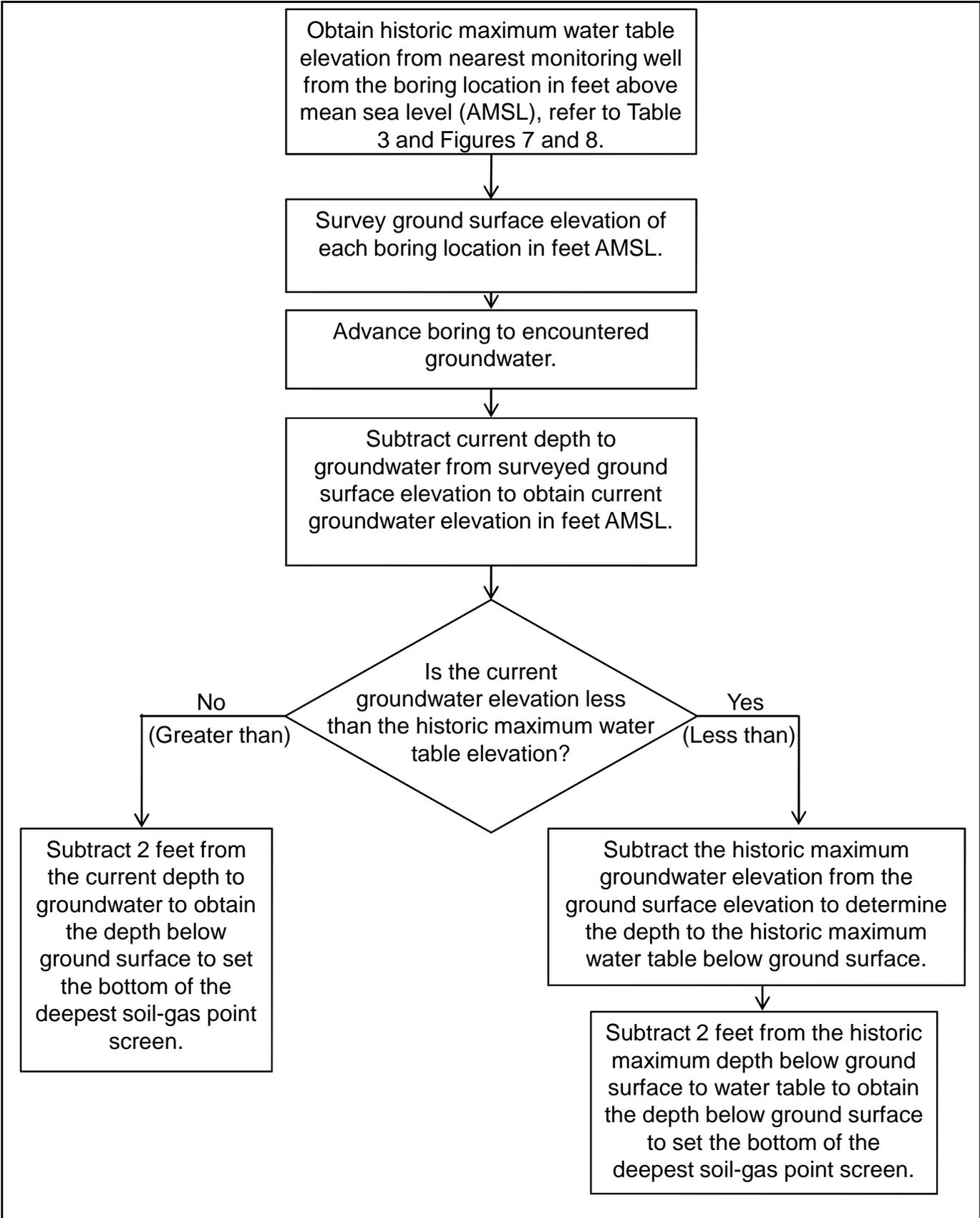
Groundwater Elevation Fluctuations  
Eastern Portion of Site  
(2006 through 2009)



FIGURE

8

Upper Aquifer Well axis is not scaled to horizontal distance between wells  
Max - maximum  
Min - minimum

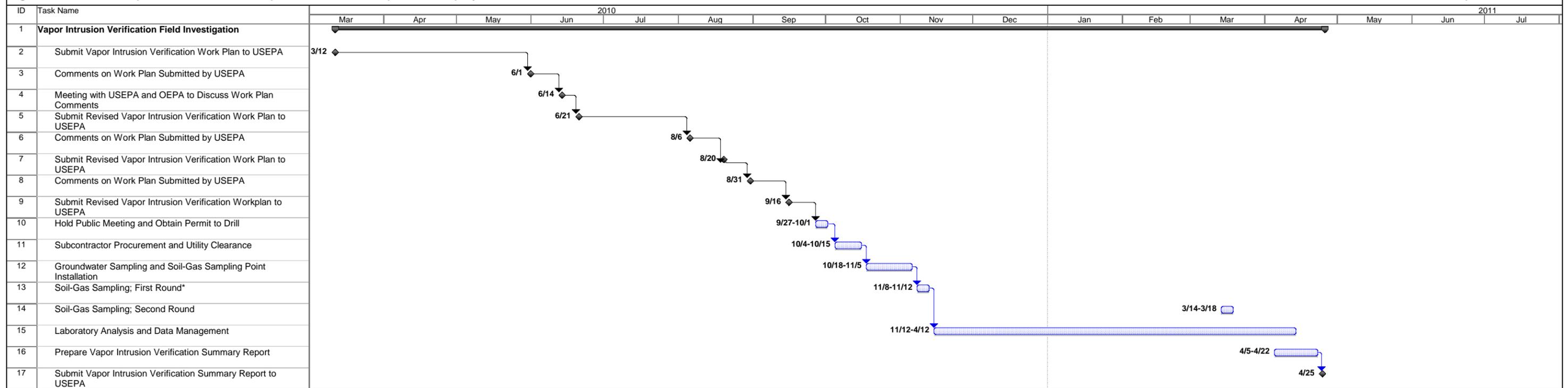


DEEPEST SOIL-GAS POINT LOCATION  
 DECISION MATRIX  
 MOTORS LIQUIDATION COMPANY  
 MORAINÉ, OHIO

Date 9/7/2010	Project Manager N. GILLOTTI	Drawing Name Deepest Soil Gas
Drawn By S. BREWER	Lead Design Prof. J. REID	Checked T. FORTNER
Project Number OH000294.0018		Figure Number 9

Figure 10. Schedule for Vapor Intrusion Verification Implementation, Motors Liquidation Company, Moraine, Ohio.

Revised September 16, 2010



Project: Revised Figure 10 VI Schedule  
Date: Fri 9/17/10

Task Milestone Summary

\* A field audit will be completed during the first round of soil-gas sampling.

## **Appendix A**

### Standard Operating Procedures

SOP 24a – Groundwater Sampling  
Using a Direct Push Sampling  
System

SOP 36 – Soil-Gas Point Installation  
and Sampling

SOP 37 – Administering Helium  
Tracer Gas for Leaks of Soil-  
Gas or Sub-slab Sampling  
Points

## **SOP 24a**

# **Groundwater Sampling Using a Direct-Push Sampling System**

Motors Liquidation Company

Moraine, Ohio

Rev. #: 2

Rev Date: August 20, 2010

**Approval Signatures**



Prepared by: \_\_\_\_\_  
Trey Fortner

Date: March 11, 2010

Revised: August 20, 2010



Reviewed by: \_\_\_\_\_  
Jason Manzo

Date: March 11, 2010

Revised: August 20, 2010

## I. Equipment List

The equipment required to install multiple nested soil vapor ports is presented below:

- Direct Push Drilling Equipment
- Paperwork
- SP16 Screen Point (4 slot)
- PPE
- Submersible Pump
- Caution Tape and Posts
- Plastic Sheeting
- Pens and Markers
- Sample Bottles
- Tubing

The direct-push unit utilizes a protected screen sampler that can sample up to approximately 4 feet of water column. To retrieve groundwater samples at the working depth of the unit a submersible pump will be used. Unlike most well points, the unit remains completely sealed by a drive point at the end of the sample tube while it is pushed or driven to the desired sampling depth.

Site specific requirements and/or field conditions may require modifications to some of the procedures outlined in this SOP. Alterations to the SOP may be completed per approval of the Project Manager.

## II. Procedure

### At Field Office:

- A. Acquire Necessary Equipment

### At Sampling Location:

1. Don appropriate PPE.
2. Establish exclusion zone with barricade tape.
3. Place plastic sheeting near work area.
4. After checking to ensure that all parts have been decontaminated, assemble groundwater sampler. All parts must fit tightly. Damage could occur during probing if threaded assemblage is not tight.

5. Drive the well point with the attached probe rods to the water table using manual probe rod driver or hydraulically-powered unit.
6. After the probe rods are driven approximately 2 feet below the water table, expose screen. Lower submersible pump and tubing down through the center of the rods into screened zone. Follow appropriate low-flow methodology for stabilization and transfer water sample to appropriate sample containers.
7. Record all details of sample collection on appropriate field forms.
8. Collect samples into appropriate bottles. Label each bottle with the following information: date, time of sampling, sample ID, analytical method, sampler initials and method of preservation. Print all information accurately and legibly. Complete chain-of-custody forms.
9. Place samples in containers as needed and pack with ice in coolers (when appropriate) as soon as possible.
10. Disassemble the sampler and remove all parts. Decontaminate equipment.
11. Personnel decontamination.

Submit samples to Sample Custodian with chain-of-custody forms and submit all paperwork

### **III. Quality Assurance**

One duplicate sample must be collected per 20 samples.

A trip blank must accompany each cooler of VOC samples that is shipped during the project.

## **SOP 36**

### **Subsurface Soil-Gas Point Installation and Sampling**

Motors Liquidation Company

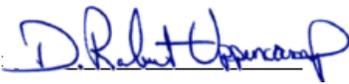
Moraine, Ohio

Rev. #: 4

Rev Date: September 10, 2010

**Approval Signatures**

Prepared by:  Date: Revised, August 20, 2010  
Mitch Wacksman

Reviewed by:  Date: May 20, 2008  
Robert Uppencamp

Approved by:  Date: Revised, October 13, 2009  
Christopher Lutes

Modified by:  Date: Revised, August 20, 2010  
Trey Fortner

Modified by:  Date: Revised, September 10, 2010  
Joseph Rumschlag

## I. Scope and Application

This document describes the procedures to collect subsurface soil-gas samples using temporary single or nested soil-gas points. Using nested soil-gas points allow for the generation of discrete data as a function of depth and time. Samples from the points are collected in an evacuated PAC250 SUMMA<sup>®</sup>-type canister, (evacuated to <28 inches of mercury [Hg]) which provides a recoverable whole-gas sample when allowed to fill to a vacuum of 2-8 inches of Hg. The whole-air sample is analyzed for volatile organic compounds (VOCs) by United States Environmental Protection Agency (USEPA) Method TO-15 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) or lower.

The following sections list the necessary equipment and provide detailed instructions for the installation of temporary single or nested soil-gas points (using direct-push technology or a hollow stem auger) and the collection of soil-gas samples for VOC analysis.

Site specific requirements and/or field conditions may require modifications to some of the procedures outlined in this standard operating procedure (SOP). Alterations to the SOP may be completed per approval of the Project Manager.

## 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 subsurface soil-gas sample collection activities must have previous subsurface soil-gas sampling experience.

## III. Health and Safety Considerations

Field sampling equipment must be carefully handled to minimize the potential for injury and the spread of hazardous substances. All sampling personnel should review the appropriate health and safety plan (HASp) and job loss analysis (JLA) prior to beginning work to be aware of all potential hazards associated with the job site and the specific installation. For subsurface soil-gas point installation, drilling with a direct-

push drilling rig or hollow stem auger rig should be done only by personnel with prior experience using such of equipment.

#### IV. Equipment List

The equipment required to install multiple nested soil gas points are 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, etc)
  - Hollow stem auger rig with interconnecting augers. The inner diameter of typical augers ranges from 2.25-inches to 7.75-inches; the auger size should be chosen to reflect the number of nested ports that will be installed inside the boring.
  - Direct-push rig (e.g., -Geoprobe®) equipped with interconnecting 4-foot lengths of steel drive rods (2.25-inch-diameter to 4-inch diameter depending on the number of ports to be installed).
- Stainless steel sample screens (one per sample depth)
- 0.25 inch outside diameter (OD) x 0.17-inch inside diameter (ID) tubing (Teflon)
- Decontaminated (cleaned) Brass valve or needle valve (one per sample depth to match sample tubing)
- Sand
- Granular bentonite (Benseal®, Volclay® Crumbles, or equivalent)
- Down hole measuring device
- Deionized, distilled, or potable water (for hydration of bentonite)
- Aluminum tags for labeling sample depths

- Flush mount well cover
- Photoionization Detector (PID) (with a lamp of 10.6 or 11.7 eV)
- Kneeling pad (as necessary)

The equipment required for soil-gas sample collection from nested soil-gas points are presented below:

- Stainless steel PAC 250 SUMMA<sup>®</sup> 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., 4 minutes, 30 minutes, 8 hours, 24 hours) or flow rate (e.g., 50 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).
- Swage-Lok fittings
- Stainless steel Swage-Lok “T” fitting (if collecting duplicate [i.e., split] samples)
- Portable vacuum pump capable of producing very low flow rates (e.g., 50 mL/min) with vacuum gauge. Purging flow rate should also be selected based on expected soil type (see below)
- Electric flow sensor (Bios DryCal or equivalent) to monitor purge rate
- Tracer gas testing supplies if applicable (refer to SOP 37 for “Administering Tracer Gas”)
- PID
- Appropriate-sized open-end wrench (typically  $\frac{9}{16}$ -inch and  $\frac{1}{2}$ -inch)
- Portable weather meter capable of collecting barometric pressure, relative humidity, and temperature, if appropriate

- Chain-of-custody (COC) forms
- Sample collection logs
- Field notebook

## V. Procedure

### Single or Nested Soil-Gas Monitoring Point Installation

The procedure used to install the single or nested soil-gas points will vary based upon the method of boring installation. In most situations a temporary well casing will need to be installed to keep the down hole formation from collapsing during point installation. The following steps will detail installing nested soil-gas points through a temporary well casing.

If the nested soil-gas points will be installed at shallow depths, or the formation is thought to be stable enough to not collapse, a temporary well casing may not be necessary to facilitate the installation of the sample points. Either way, the steps for installing the sample points are nearly identical. These following steps should be discussed with the drilling subcontractor and altered based on the methods chosen for a given project.

1. Advance boring to bottom of deepest sampling interval and install a temporary well casing. Soil-gas points will not be installed in groundwater or the capillary fringe. Moisture conditions and/or other observations (such as depth to water in nearby monitoring wells) should be recorded on the soil-gas collection log, as indicated.
2. Fill in 2 inches of sand pack for below the soil-gas point and measure to make sure that the total depth is correct for the stainless steel sample screen to be installed.
3. Cut a length of sample collection tubing slightly longer (e.g., 4 to 5 feet) than the collection depth. Attach a stainless steel sample screen to one end of the sample collection tubing and lower the screen and attached tubing through the boring.

4. Assure that the sample screen has reached the bottom of the boring and record this depth.
5. Begin simultaneously filling in the area around the sample screen with clean sand and retracting the temporary well casing. The casing should be lowered back down onto the sand every few inches to compact the sand around the screen. Sand should be introduced to cover the sample screen (6 inches deep) then to extend another six inches above the screen for a total of 14 inches of sand.
6. With the proper sand pack in place begin slowly introducing granular bentonite to the boring while gradually retracting the well casing. A minimum of 12-inches of dry granular bentonite should be added followed by hydrated bentonite to the next sample depth (for nested points) or to the ground surface (for single points). This dry bentonite interval will help ensure water does not reach the sand pack around the sample screen.
7. Properly label the sample tubing with a permanent label to designate the sample number and screen depth with aluminum tags.
8. Affix a Swagelok brand or equivalent fitting and valve to the end of the tubing. Use caution to not interchange fittings from different manufacturers as this may result in a compromised seal and possible leakage.
9. Repeat steps 2-8 until all the sample depths are installed.
10. When installing the shallowest (last) sample point, bentonite should be emplaced all the way to the ground surface or bottom of the vault (if applicable) for a minimum of 3 feet of bentonite to ensure no short circuiting with the ambient air
11. With all temporary single or nested points installed and labeled, a well cover may be installed.
  - a. For permanent installations, the well cover should be rated for whatever type of traffic it may encounter in the future. For interior installations a brass clean-out cover available from a plumbing supply store may provide adequate protection. For exterior installations in high traffic areas a heavy duty groundwater well cover may be appropriate.

- b. For a temporary installation, a well cover is generally not necessary as the tubing will be removed within several days.
12. All soil-gas points should be allowed to sit and equilibrate for a minimum of 48 hours before proceeding to soil-gas sample collection (CEPA, 2010).

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

The following steps should be used to collect a soil-gas sample from each of the single or nested points installed using the above procedure.

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 (if capable with in-field measuring device);
  - b. ambient temperature;
  - c. barometric pressure; and
  - d. relative humidity.
2. Connect a properly calibrated portable vacuum pump to the sample tubing.

3. Purge 3 volumes of air from the soil-gas point screen and sampling line using a portable pump at a rate of approximately 50 mL/min. Calculate three-times the volume of the inside of the sample tubing, sample screen, sand pack and dry bentonite seal using the calculation:

$$V_1 + V_2 + V_3 = V_t$$

where:

$V_1 = \pi r^2 h$  = open space volume of soil-gas screen

$V_2 = \pi r^2 h$  = open space volume of sample tubing

$V_3 = \pi r^2 h \rho$  = estimated volume of sand pack and dry bentonite open pore space

$V_t$  = total volume

$r$  = inner radius of soil-gas screen, borehole, or sample tubing

$h$  = height of soil-gas screen or height (length) of tubing or height of sand pack and dry bentonite seal

$\rho$  = porosity of sand pack and dry bentonite (40%)

The 50 mL/min flow rate should be suitable for a variety of silt and sand conditions but will not be achievable in some clays without excessive vacuum. Excessive vacuums should be avoided. A low vacuum (<10 inches Hg) should be maintained. Record the measured flow rate and vacuum pressure during sample collection.

The cutoff value for vacuum differs in the literature from 10 inches of water column (CEPA, 2010) to 136 inches of water column or 10 inches of Hg ([http://www.dtsc.ca.gov/SiteCleanup/upload/SAG\\_Review\\_Drft](http://www.dtsc.ca.gov/SiteCleanup/upload/SAG_Review_Drft)). A detailed discussion of the achievable flow rates in various permeability materials can be found in Nicholson 2007. Related issues of contaminant partitioning are summarized in ASTM D5314-92. Passive sampling approaches can be considered as an alternative for clay soils. Measure organic vapor levels with the PID.

4. All SUMMA ®-type canisters received from Air Toxics will be checked for correct vacuum. The vacuum gauges provided by the analytical laboratory as part of the sample train (i.e., canister and flow controller) are used to record the initial and final vacuums in the air sampling canister. Pre-sampling vacuum in the canister should be between -30 inches of mercury (in Hg) and -25 in Hg. In the event a canister is not within this initial range, it will be rejected and a new canister, flow controller and vacuum will be similarly checked.

5. If low-flow conditions are encountered (when air flow rates are less-than 10 mL/min or when vacuum is greater than 10 inches of Hg) and preclude the collection of representative soil-gas samples, due to high moisture conditions and/or tight soils, a replacement probe should be installed approximately 5 feet from the original location, for up to 3 attempts.
6. A “shut in” test will be preformed prior to sampling each soil-gas sample point to test the integrity of all above ground sampling equipment supplied by the laboratory (i.e., SUMMA® canister, flow controller, vacuum gauge, and associated fittings). All above ground sampling equipment will be assembled and the cap from the SUMMA® canister will be placed on the end of the sample train, effectively creating a closed system. The SUMMA® canister valve will then be briefly opened then closed; the vacuum applied by the canister is then effectively “shut-in” to the sample train. The vacuum gauge will be observed for at least one minute, and if there is any appreciable loss in vacuum, fittings should be adjusted to remedy the situation and create a leak-free environment. In the event a leak cannot be remedied, field staff should reject the sampling apparatus and choose another unit.
7. A tracer gas leak test should be conducted to ensure that ambient leakage is either not occurring or is within acceptable limits. Check the seal established around the all soil-gas points by using a tracer gas (e.g., helium) or other method established in the state guidance documents. [Note: Refer to SOP “Administering Tracer Gas,” for procedures on tracer gas use.] If unacceptable leaks are detected ( $\geq 5\%$  of the source concentration), take corrective action to seal all potential sources of leak in the sampling train. If the problem cannot be corrected, a replacement probe should be installed and sampled approximately 5 feet from the original location.
8. Remove the brass 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.
9. Connect the Teflon sample collection tubing to the flow controller and the SUMMA® canister valve.

10. Open the SUMMA® canister valves. Record in the field notebook and/or sample log (attached) the time sampling began, the canister vacuum (as noted on the vacuum gauge), the sampling flow rate specified from the laboratory, canister serial number, and flow controller serial number.
11. Take photographs of the SUMMA® canister and surrounding area; as appropriate.
12. Steps 2-10 should be repeated for each of the nested soil-gas points; samples can be collected concurrently.

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

1. Due to the short duration of the soil-gas samples, field staff should stay at the SUMMA® canister location through the entire sampling interval.
2. Record the final canister vacuum-. Stop collecting the sample by closing the SUMMA® canister valves. The canister should have a minimum amount of vacuum (approximately 5-inches of Hg or slightly greater). The duration of a PAC250 canister collecting at 50 mL/min and leaving 5-inches of Hg residual vacuum in the canister is approximately 4-minutes.
3. Record the date and time of valve closing in the field notebook, sample collection log, and COC form.
4. Close the valve on the nested soil-gas sample tubing.
5. Once all the nested samples have been collected, be sure the well cover (if applicable) is properly re-installed and secured.
6. Remove the particulate filters and flow controllers from the SUMMA® canisters, re-install the brass plugs on the canister fittings, and tighten with the appropriate wrench.
7. Package the canisters and flow controllers in the shipping container supplied by the laboratory for return shipment to the laboratory. The SUMMA® canisters should not be preserved with ice or refrigeration during shipment.

8. Complete the appropriate forms and sample labels as directed by the laboratory (e.g., affix card with a string).
9. 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 carrier (e.g., Federal Express) for analysis.
10. In accordance with standard operating procedures, equipment decontamination procedures, chain-of-custody procedures, and equipment calibration and maintenance procedures should be strictly followed.

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

If the single or nested soil-gas points were installed in a temporary manner, and the soil-gas samples have been collected, the soil-gas monitoring points will be abandoned by pulling up the sample tubing. Since the boring is filled with bentonite and sand, no additional abandonment steps are necessary. Ensure that the boring location and surrounding area are returned to as close to their original appearance as possible.

#### **VII. Cautions**

The following cautions and field tips should be reviewed and considered prior to installing or collecting a single or nested soil-gas sample.

- 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 be taken to ensure that the flow controller is pre-calibrated to the proper sample collection time (confirm with laboratory prior to sampling event, and confirm on packaging list). 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. Excessive vacuum remaining in the canister can also result in elevated reporting limits.

- Care should be taken to ensure that nested ports are installed at the target sample depths. Sampling personnel should work closely with the driller to ensure this is implemented.
- When introducing granular bentonite to the boring, the material should be introduced slowly and hydrated properly. Consult the bentonite manufacturer's instructions on the bag to determine the proper amount of to be used. When hydrated properly bentonite forms a thick clay mass that remains moist. The hydration step is crucial in the installation process and if not done properly the integrity of the bentonite seal can be compromised.
- Installing a layer of dry bentonite directly above the sand pack will help ensure water does not reach the sand pack around the sample screen.
- It is important to record the canister pressure, start and stop times and ID on a proper field sampling form. Often SUMMA® canisters are collected with a 24 hour averaging period. You should observe and record the time/pressure at the start, and then again one or two hours after starting the sample collection. It is a good practice to lightly tap the pressure gauge with your finger before reading it to make sure it isn't stuck. If the canister is running correctly for a 24 hour period then the vacuum will have decreased slightly after an hour or two (for example from 29 inches to 27 inches). Consult your Project Manager (PM), risk assessor or air sampling expert by phone if the SUMMA® canister does not appear to be working properly.
- Ensure that there is still measureable vacuum in the SUMMA® after sampling. In some cases the gauges sent from labs may have large offset errors, or they malfunction (stick). For the most precise pressure readings consider using a separate, more sensitive, device to do checks at the beginning and end of the sampling period. If this is used, it must be tested beforehand to confirm that it does not introduce contaminants to the can during pressure checks.
- If possible, have equipment shipped a day or two before the sampling date so that all materials can be checked.
- Requesting extra canisters from the laboratory should also be considered to ensure that you have enough equipment on site in case of an equipment failure.

- Soil-gas sampling should not proceed within 5 days following a significant rain event (1/2-inch of rainfall or more).

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.

### **VIII. Waste Management**

The waste materials generated by these activities should be minimal. Personal protective equipment, such as gloves and other disposable equipment (i.e., tubing) should be collected by field personnel for proper disposal. Any soils brought up from the borehole should be disposed of in a manner consistent with the project work plan.

### **IX. Data Recording and Management**

Measurements will be recorded in the field notebook and/or sample log (attached) 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, flow rate, initial vacuum reading, and final vacuum reading. Field sampling logs and COC records will be transmitted to the Project Manager.

### **X. Quality Assurance**

Duplicate samples should be collected in the field as a quality assurance step. Duplicate samples will be collected at a rate of 1 per 20 air samples (5%).

Soil-gas sample analysis will generally be performed using USEPA TO-15 methodology or a project specific constituent list. Method TO-15 uses a quadrupole or ion-trap GC/MS with a capillary column to provide optimum detection limits (typically 0.5-ppbv for most VOCs prior to any dilution). Duplicate soil gas samples should be collected via a split sample train, allowing the primary and duplicate sample to be collected from the soil-gas probe simultaneously.

Trip blank samples will not be used during soil gas sampling. SUMMA® canisters are self-sealed containers which do not permit any contamination to enter during shipment or storage. Furthermore all parts of the SUMMA® canister are metal and non-porous, therefore there is no potential for any contamination to be absorbed. The batch

certified clean SUMMA® canisters will be provided by the laboratory. The only potential contamination would come from a possible leak in the SUMMA® canister. The integrity of each SUMMA® canister will be confirmed prior to sampling by measuring the vacuum within the canister, with follow up measurements after the canister is filled in the field, and upon arrival at the laboratory.

## XI. References

ASTM – “Standard Guide for Soil Gas Monitoring in the Vadose Zone”, D5314-92.

Hayes, H. C., D.J. Benton and N. Khan “Impact of Sampling media on Soil Gas Measurements”  
Presented with short paper at AWMA Vapor Intrusion Conference January 2006,  
Philadelphia PA.

CEPA, 2010: California Environmental Protection Agency. 2010. “Advisory – Active Soil  
Investigation”. March.

[http://www.dtsc.ca.gov/SiteCleanup/upload/SAG\\_Review\\_Drft.pdf](http://www.dtsc.ca.gov/SiteCleanup/upload/SAG_Review_Drft.pdf)

ITRC “Vapor Intrusion Pathway: A Practical Guide”, January 2007, Appendix F: “regulators  
Checklist for Reviewing Soil Gas Data”.

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

Nicholson, P, D. Bertrand and T. McAlary. “Soil Gas Sampling in Low-Permeability Materials”  
Presented at AWMA Specialty Conference on Vapor Intrusion, Providence RI, Sept.  
2000.



## Sub-slab/Soil-Gas Sample Collection Log

		Sample ID:	
Client:		Boring Equipment:	
Project:		Sealant:	
Location:		Tubing Information:	
Project #:		Miscellaneous Equipment:	
Samplers:		Subcontractor:	
		Equipment:	
Sampling Depth:		Moisture Content of Sampling Zone):	
Time and Date of Installation:		Approximate Purge Volume:	

### Instrument Readings:

Date	Time	Canister Vacuum (a) (inches of Hg)	Temperature (°F)	Relative Humidity (%)	Air Speed (mph)	Barometric Pressure (inches of Hg)	PID (ppb)

(a) Record canister information at a minimum at the beginning and end of sampling

#### SUMMA® Canister Information:

Size (circle one):	250 mL	1L
Canister ID:		
Flow Controller ID:		
Notes:		

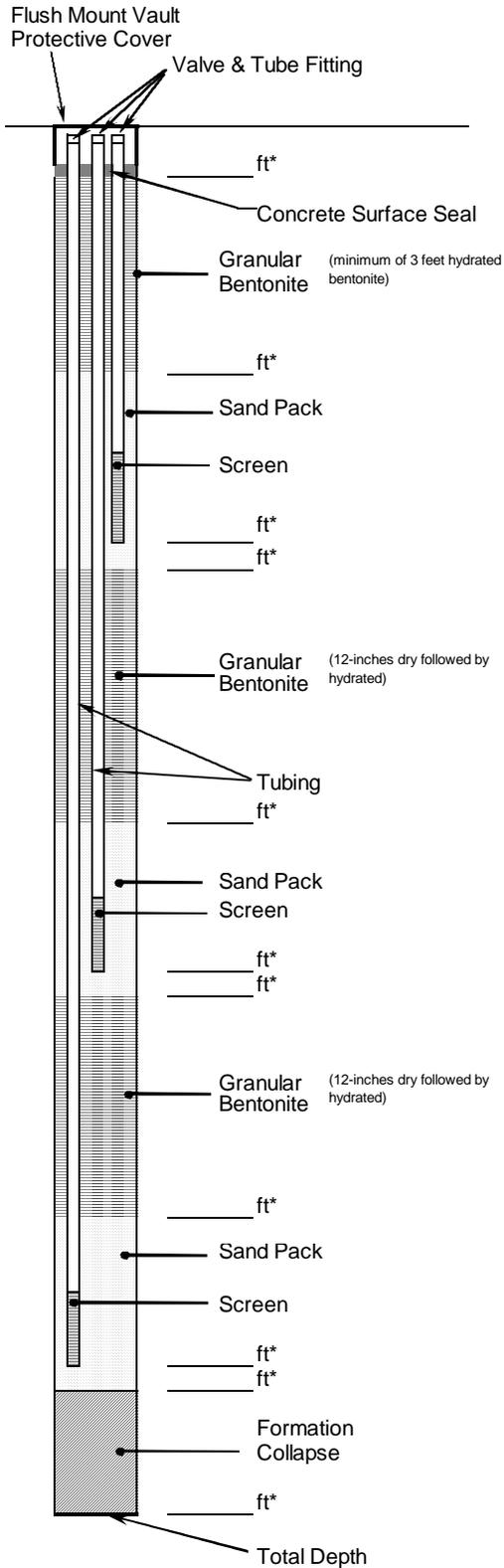
#### Tracer Test Information (if applicable):

Initial Helium Shroud:		
Final Helium Shroud:		
Tracer Test Passed:	Yes	No
Notes:		

### General Observations/Notes:


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

$V_1 + V_2 + V_3 = V_t$  where:  $V_1 = \pi r^2 h$  = open space volume of soil-gas screen;  $V_2 = \pi r^2 h$  = open space volume of sample tubing;  $V_3 = \pi r^2 h \rho$  = estimated open pore space for sand pack and dry bentonite seal;  $V_t$  = total volume;  $r$  = inner radius of soil-gas screen, borehole, or sample tubing;  $h$  = height of soil-gas screen or height (length) of tubing or height of sand pack and dry bentonite seal;  $\rho$  = porosity of sand pack and dry bentonite seal (40%).



\* Depth in Feet Below Land Surface

Project: \_\_\_\_\_ Point: \_\_\_\_\_

City: \_\_\_\_\_

County: \_\_\_\_\_ State: \_\_\_\_\_

Survey Coordinates:

Northing: \_\_\_\_\_

Easting: \_\_\_\_\_

Land-Surface Elevation (surveyed): \_\_\_\_\_ feet  
(above mean sea level)

Historic Maximum Groundwater Elevation: \_\_\_\_\_ feet  
(above mean sea level)

Groundwater Encountered During Drilling: \_\_\_\_\_ feet bls

Installation Dates: \_\_\_\_\_

Weather Conditions at Installation: \_\_\_\_\_

Drilling Contractor: \_\_\_\_\_

Driller: \_\_\_\_\_

Drilling Method: \_\_\_\_\_

Screen Construction: Type: Stainless Steel  
Length: 6 inches  
Diameter: 0.4375-inch OD, 0.3125-inch ID

Tubing Construction: Type: Teflon  
Diameter: 0.25-inch OD, 0.17-inch ID

Volume Calculation (mL):	Shallow	Middle	Deep
$V_1$	_____	_____	_____
$V_2$	_____	_____	_____
$V_3$	_____	_____	_____
$V_t$	_____	_____	_____

Remarks: \_\_\_\_\_

Prepared by: \_\_\_\_\_

$V_1 + V_2 + V_3 = V_t$  where:  $V_1 = \pi r^2 h$  = open space volume of soil-gas screen;  $V_2 = \pi r^2 h$  = open space volume of sample tubing;  $V_3 = \pi r^2 h \rho$  = estimated open pore space for sand pack and dry bentonite seal;  $V_t$  = total volume;  $r$  = inner radius of soil-gas screen, borehole, or sample tubing;  $h$  = height of soil-gas screen or height (length) of tubing or height of sand pack and dry bentonite seal;  $\rho$  = porosity of sand pack and dry bentonite seal (40%).

OD - outer diameter                      12-inches = 1 foot  
ID - inner diameter                        1 cubic inch = 16.39 mL  
bls - below land surface                     $\pi = 3.1416$

## **SOP 37**

### **Administering Helium Tracer Gas for Leak Checks of Soil-Gas or Sub-slab Sampling Points**

Motors Liquidation Company

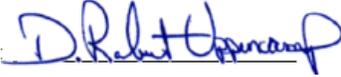
Moraine, Ohio

Rev. #: 2

Rev Date: August 20, 2010

**Approval Signatures**

Prepared by:  Date: May 20, 2008  
Mitch Wacksman

Reviewed by:  Date: May 20, 2008  
Robert Uppencamp

Approved by:  Date: November 14, 2008  
Christopher Lutes

Modified by:  Date: Revised August 20, 2010  
Trey Fortner

## I. Scope and Application

When collecting subsurface soil-gas samples as part of a vapor intrusion evaluation, a tracer gas serves as a quality assurance/quality control device to verify the integrity of the soil-gas point seal. Without the use of a tracer, verification that a soil-gas sample has not been diluted by ambient or indoor air is difficult.

This standard operating procedure (SOP) focuses on using helium as a tracer gas. However, depending on the nature of the contaminants of concern, other compounds can be used as a tracer including sulfur hexafluoride (SF<sub>6</sub>), butane and propane (or other gases). In all cases, the protocol for using a tracer gas is consistent and includes the following basic steps: (1) enrich the atmosphere in the immediate vicinity where the port or sample tubing intersects the surface with the tracer gas; and (2) measure a vapor sample from the sample tubing for the presence of high concentrations (>5%) of the tracer. A pail, bucket, garbage can or even a plastic bag can serve to keep the tracer gas in contact with the port during the testing.

There are two basic approaches to testing for the tracer gas:

1. Include the tracer gas in the list of target analytes reported by the laboratory; or
2. Use a portable monitoring device to analyze a sample of soil-gas for the tracer prior to sampling for the compounds of concern. (Note that tracer gas samples can be collected via syringe, Tedlar bag, etc. They need not be collected in SUMMA® canisters or minicans.)

This SOP focuses on monitoring helium using a portable sampling device, although helium can also be analyzed by the laboratory along with other volatile organic compounds (VOCs). Real-time tracer sampling is generally preferred as the results can be used to confirm the integrity of the port seals prior to formal sample collection.

During the initial stages of a subsurface soil-gas sampling program, tracer gas samples should be collected at each of the sampling points or in the case of nested points in the shallowest sampling point. If the results of the initial samples indicate that the port seals are adequate, the Project Manager can consider reducing the number of locations at which tracer gas samples are used. At a minimum, at least 10% of the subsequent samples should be supported with tracer gas analyses. When using permanent soil-gas points as part of a long-term monitoring program, the port should

be tested prior to the first sampling event. Tracer gas testing of subsequent sampling events is not necessary unless conditions have changed at the site.

Site specific requirements and/or field conditions may require modifications to some of the procedures outlined in this SOP. Alterations to the SOP may be completed per approval of the Project Manager.

## **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 the tracer gas testing must have previous experience conducting similar tests.

## **III. Equipment List**

The equipment required to conduct a helium tracer gas test are presented below:

- Appropriate PPE for site (as required by the Health and Safety Plan)
- Helium (laboratory grade)
- Regulator for helium tank
- Shroud (plastic bucket, garbage can, etc)
  - The size of the shroud should be sufficient to fit over the soil-gas point manhole. It is worth noting that using a smaller shroud obviously uses less helium as well; this may be important when projects require a number of helium tracer tests.
  - The shroud will need to have three small holes in it. These holes will include one on the top (to accommodate the sample tubing), and two on the side (one for the helium detector probe, and one for the helium line).

- The shroud ideally encloses the entire sampling train.
- Helium detector capable of measuring from 1 - 100% (Dielectric MGD-2002, Mark Model 9522, or equivalent)
- Tedlar bags
- Seal material for shroud (rubber gasket, modeling clay, bentonite, etc).  
Although the sealing material is not in direct contact with the sample if no leak occurs, sealing materials with high levels of VOC emissions should be avoided, since they could easily contaminate a sample from a point in which a trace leak occurs.
- Field notebook

#### IV. Procedure

The procedure used to conduct the helium tracer test should be specific to the shroud being used and the methods of soil-gas point installation. The helium tracer test can be conducted when using temporary or permanent sample point installs and from inside or outside a facility. However, when using the tracer gas within an indoor area you must provide adequate ventilation because helium is an asphyxiant.

1. Attach Teflon sample tubing to the sample point. This can be accomplished utilizing a number of different methods depending on the sample install (i.e., barbed fitting, Swage-Lok fitting, ball valve, etc.).
2. Place the shroud over the sample point and tubing.
3. Pull the tubing through hole in top of shroud. Seal opening with modeling clay.
4. Place weight on top of shroud to help maintain a good seal with the ground.
5. Insert helium tubing into hole in side of shroud, seal with modeling clay to prevent leaks.
6. Fill shroud with helium. While filling shroud allow atmospheric air to escape either by leaving a crack with the surface or by providing a release valve on the side of the shroud.

7. Use the helium detector to test level of helium gas from the bottom of the shroud (where the sample tubing intersects the ground). Helium should be added until the environment inside the shroud has > 60% helium.
8. Purge the sample point through the sample tubing into a Tedlar bag using a hand held sampling pump. The sample pump should be operating at a rate of 50 mL/minute (the purge rate should not exceed the sample collection rate). Use a stand-alone flow sensor to monitor purge flow rate during purge (Bios DryCal or equivalent). Test the air in the Tedlar bag for helium using portable helium detector. If the sample point has been installed properly there should be zero helium in purge air.
9. If > 5% helium is noted in purge air, add more clay or other material to the seal the sample port at the surface and repeat the testing procedure. If the seal cannot be fixed, re-install sample point.
10. Monitor and record helium level in shroud before, during and after tracer test.
11. Monitor and record helium level in purge exhaust.
12. At successful completion of tracer test and sample point purging, the soil-gas sample can be collected (if the helium shroud must be removed prior to sample collection be mindful not disturb the sample tubing and any established seals).

#### **V. Cautions**

Helium is an asphyxiant! Be cautious with its use indoors!

Care should be taken not to pressurize shroud while introducing helium. If the shroud is completely air tight and the helium is introduced quickly, the shroud can be over-pressurized and helium can be pushed into the ground.

Because minor leakage around the port seal should not materially affect the usability of the soil-gas sampling results, the mere presence of the tracer gas in the sample should not be a cause for alarm. Consequently, portable field monitoring devices with detection limits in the low ppm range are more than adequate for screening samples for the tracer. If high concentrations (>5%) of tracer gas are observed in a sample, the port seal should be enhanced to reduce the infiltration of ambient air and the tracer

test readministered. If the problem cannot be rectified, a new sample point should be installed.

## **VI. 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, and the helium concentrations in both the shroud and the purge air before, during, and after tracer testing. Any problems encountered should also be recorded in the field notes.

**APPENDIX: Compressed Gases—Use and Storage**

In general, a compressed gas is any material contained under pressure that is dissolved or liquefied by compression or refrigeration. Compressed gas cylinders should be handled as high-energy sources and therefore as potential explosives and projectiles. Prudent safety practices should be followed when handling compressed gases since they expose workers to both chemical and physical hazards.

**Handling**

- Safety glasses with side shields (or safety goggles) and other appropriate personal protective equipment should be worn when working with compressed gases.
- Cylinders should be marked with a label that clearly identifies the contents.
- All cylinders should be checked for damage prior to use. Do not repair damaged cylinders or valves. Damaged or defective cylinders, valves, etc., should be taken out of use immediately and returned to the manufacturer/distributor for repair.
- All gas cylinders (full or empty) should be rigidly secured to a substantial structure at 2/3 height. Only two cylinders per restraint are allowed in the laboratory and only soldered link chains or belts with buckles are acceptable. Cylinder stands are also acceptable but not preferred.
- Handcarts shall be used when moving gas cylinders. Cylinders must be chained to the carts.
- All cylinders must be fitted with safety valve covers before they are moved.
- Only three-wheeled or four-wheeled carts should be used to move cylinders.
- A pressure-regulating device shall be used at all times to control the flow of gas from the cylinder.
- The main cylinder valve shall be the only means by which gas flow is to be shut off. The correct position for the main valve is all the way on or all the way off.
- Cylinder valves should never be lubricated, modified, forced, or tampered with.
- After connecting a cylinder, check for leaks at connections. Periodically check for leaks while the cylinder is in use.
- Regulators and valves should be tightened firmly with the proper size wrench. Do not use adjustable wrenches or pliers because they may damage the nuts.

- Cylinders should not be placed near heat or where they can become part of an electrical circuit.
- Cylinders should not be exposed to temperatures above 50 °C (122 °F). Some rupture devices on cylinders will release at about 65 °C (149 °F). Some small cylinders, such as lecture bottles, are not fitted with rupture devices and may explode if exposed to high temperatures.
- Rapid release of a compressed gas should be avoided because it will cause an unsecured gas hose to whip dangerously and also may build up enough static charge to ignite a flammable gas.
- Appropriate regulators should be used on each gas cylinder. Threads and the configuration of valve outlets are different for each family of gases to avoid improper use. Adaptors and homemade modifications are prohibited.
- Cylinders should never be bled completely empty. Leave a slight pressure to keep contaminants out.

#### **Storage**

- When not in use, cylinders should be stored with their main valve closed and the valve safety cap in place.
- Cylinders must be stored upright and not on their side. All cylinders should be secured.
- Cylinders awaiting use should be stored according to their hazard classes.
- Cylinders should not be located where objects may strike or fall on them.
- Cylinders should not be stored in damp areas or near salt, corrosive chemicals, chemical vapors, heat, or direct sunlight. Cylinders stored outside should be protected from the weather.

#### *Special Precautions*

#### Flammable Gases

- No more than two cylinders should be manifolded together; however several instruments or outlets are permitted for a single cylinder.
- Valves on flammable gas cylinders should be shut off when the laboratory is unattended and no experimental process is in progress.
- Flames involving a highly flammable gas should not be extinguished until the source of the gas has been safely shut off; otherwise it can reignite causing an explosion.

#### Acetylene Gas Cylinders

- Acetylene cylinders must always be stored upright. They contain acetone, which can discharge instead of or along with acetylene. Do not use an acetylene cylinder that has been stored or handled in a nonupright position until it has remained in an upright position for at least 30 minutes.
- A flame arrestor must protect the outlet line of an acetylene cylinder.
- Compatible tubing should be used to transport gaseous acetylene. Some tubing like copper forms explosive acetylides.

#### Lecture Bottles

- All lecture bottles should be marked with a label that clearly identifies the contents.
- Lecture bottles should be stored according to their hazard classes.
- Lecture bottles that contain toxic gases should be stored in a ventilated cabinet.
- Lecture bottles should be stored in a secure place to eliminate them from rolling or falling.
- Lecture bottles should not be stored near corrosives, heat, direct sunlight, or in damp areas.
- To avoid costly disposal fees, lecture bottles should only be purchased from suppliers that will accept returned bottles (full or empty). Contact the supplier before purchasing lecture bottles to ensure that they have a return policy.
- Lecture bottles should be dated upon initial use. It is advised that bottles be sent back to the supplier after one year to avoid accumulation of old bottles.

ARCADIS

**Appendix B**

Laboratory Methodology and QA/QC

DLZ Ohio, Incorporated

Air Toxics Limited

TestAmerica

## Introduction

As an amendment to the draft Quality Assurance Project Plan (QAPP) dated December 23, 2003, the presented information outlines quality assurance/quality control (QA/QC) protocols that will be followed for geotechnical, groundwater, and soil-gas samples for the Vapor Intrusion Verification Work Plan.

## Geotechnical Samples

As discussed in the Vapor Intrusion Verification Work Plan, geotechnical samples will be taken at several locations at the Site. The laboratory utilized will be DLZ Ohio, Inc. located in Columbus, Ohio. Table B-1 provides a summary of the parameters, containers, packaging, shipping, and analytical methods. Attachment B-1 provides DLZ Construction Materials Laboratory and Construction Monitoring and Testing Services Quality Assurance/Quality Control Manual dated January 14, 2010 and Consolidated Soil Indexing Formulas to be used in the air filled/water filled porosity calculation dated 1997.

## Soil-Gas Samples

As discussed in the Vapor Intrusion Verification Work Plan, soil-gas samples will be collected at the Site. The laboratory utilized will be Air Toxics Limited located in Folsom, California. Table B-1 provides a summary of the parameters, containers, packaging, shipping, and analytical methods. Attachment B-2 provides Air Toxics Limited Methods Manual, Revision 17.1, December, 2009. This manual provides specifics on methodologies used to analyze soil-gas samples. Air Toxics Laboratory employs a true batch certification for the PAC250 SUMMA® canisters (10%).

Air Toxics is certified in the National Environmental Laboratory Accreditation Program (NELAP). Air Toxics' primary NELAP certification number as issued by the Florida Department of Health is E87680. Air Toxics provides a quality assurance section on their website (<http://www.airtoxics.com/cinfo/qc.html>) and also has a laboratory quality assurance program. Specific Air Toxics standard operating procedures (SOPs) that will be utilized during soil-gas sampling are as follows:

- USEPA method TO-15: SOP #6-Revision 25, 6/27/2010
- Canister cleaning and preparation: SOP #7-Revision 25, 8/20/2009
- Flow controller preparation: SOP #70-Revision 6, 6/27/2010

Air Toxics Laboratory's SOPs are proprietary and are not available for public distribution. If requested, Air Toxics Laboratory would make this SOP available to the EPA with a non-disclosure agreement for access via a secure Web Portal. Please contact Ausha Scott with Air Toxics Laboratory for more information (916-605-3344).

Trip Blanks will not be submitted with soil-gas samples. Trip blanks assess potential sample contamination resulting from the transportation and storing of samples." In the case of utilizing SUMMA® canisters to sample soil-gas, this statement is inappropriate. SUMMA® canisters are self-sealed containers which would not permit any contamination to enter during shipment or storage. Furthermore all parts of the SUMMA® canister are metal and non-porous, therefore there is no potential for any contamination to be absorbed. The certified clean SUMMA® canisters will be provided by the laboratory. The only potential contamination would come from a possible leak in the SUMMA® canister. The integrity of each SUMMA® canister will be confirmed prior to sampling by measuring the pressure within the canister, with follow up measurements after the canister is filled in the field as well as upon arrival at the laboratory. Based on this, the SOP 36 has been revised and the information regarding trip blanks removed. Trip blanks are not proposed to be submitted during the field work associated with the Work Plan. Table B-3 summarizes the QC samples to be collected for soil-gas samples.

## **Groundwater Samples**

As discussed in the Vapor Intrusion Verification Work Plan, groundwater samples will be collected at the Site. The laboratory utilized for analysis of groundwater samples will be TestAmerica Analytical Testing Corporation located in North Canton, Ohio. Table B-1 provides a summary of the parameters, containers, packaging, shipping, and analytical methods. Table B-2 provides the site-specific list of volatile organic compounds and estimated quantitation limits from TestAmerica Analytical Testing Corporation, North Canton, Ohio. Table B-3 summarizes the QC samples to be collected for groundwater samples. Attachment B-3 provides TestAmerica Quality Assurance Manual, Revision 0, January 1, 2008; Determination of Volatile Organics by GS/MS based on Methods 8260B and 8260A, Revision 1, January 7, 2009; Corporate Management Plan, Revision 0, January 12, 2009; Laboratory Reference Data for site specific list of VOCs, June 16, 2010; and letter from TestAmerica to the Illinois Environmental Protection Agency explaining the laboratory purchase of Severn Trent Laboratories dated June 22, 2007.

## ARCADIS

Table B-1. Sample Container, Preservation, Shipping, and Packaging Requirements, Motors Liquidation Company, Moraine, Ohio.

*Air Toxics Limited, Folsom, California*

Target Parameters	Analytical Method	Containers, Preservatives, Holding Times Air	Laboratory SOP Reference
Site-Specific VOCs	USEPA TO-15	Evacuated stainless steel canister - SUMMA® No Preservative Hold time - 30 days prior and 30 days after sampling	Air Toxics Limited Methods Manual Revision 17.1, December 2009

*DLZ Ohio, Inc. Columbus, Ohio*

Target Compounds	ASTM	Containers, Preservatives, Holding Times Air	Laboratory SOP Reference
Moisture Content Specific Gravity Particle-Size Analysis Permeability Air Filled/Water Filled Porosity	D 2216 D 854 D 422 D 2434, D 5084 NA	Thin Walled Sampling Tube No Preservative No Hold Time	DLZ Construction Materials Laboratory and Construction Monitoring and Testing Services Quality Assurance/Quality Control Manual January 14, 2010

*TestAmerica Analytical Testing Corporation, North Canton, Ohio*

Target Compounds	Analytical Method	Containers, Preservatives, Holding Times Air	Laboratory SOP Reference
Site-Specific VOCs	USEPA Method 8260B	40 milliliter glass vial Hydrochloric acid preservative Hold time - 14 days from sampling to analysis	SOP No. NC-MS-019, Rev. 1 Determination of Volatile Organics by GC/MS based on Methods 8260B and 8260A January 7, 2009

NA - not applicable; calculations based on formulas derived from soil physical properties (Appendix B - DLZ Consolidated Soil Indexing Formulas).

VOCs - Volatile organic compounds.

U.S. EPA - United States Environmental Protection Agency.

SOP - Standard operating procedure.

GC/MS - Gas chromatography-mass spectrometry.

# ARCADIS

**Table B-2. Site-Specific VOC Parameter List and EQLs for TestAmerica Analytical Testing Corporation of North Canton; Ohio, Motors Liquidation Company, Moraine, Ohio.**

Parameter List	Water	
	Unit	EQL
<b>Site-Specific VOCs</b>		
cis-1,2-Dichloroethene	µg/L	1
trans-1,2-Dichloroethene	µg/L	1
Benzene	µg/L	1
Ethylbenzene	µg/L	1
Tetrachloroethene	µg/L	1
Toluene	µg/L	1
Trichloroethene	µg/L	1
Vinyl chloride	µg/L	1
Xylenes (total)	µg/L	2
1,1-Dichloroethane	µg/L	1
1,1-Dichloroethene	µg/L	1
1,1,1-Trichloroethane	µg/L	1

VOCs - Volatile Organic Compounds.

EQL - Estimated Quantitation Limits provided by TestAmerica Analytical Testing Corporation, North Canton, Ohio.

µg/L - Micrograms per liter.

**Table B-3. Internal Quality Control Checks, Motors Liquidation Company, Moraine, Ohio.**

Description of Soil-Gas Samples	Number of Samples	Field QC Samples	Laboratory QC Methods <sup>(2)</sup>
Soil-gas sampling points will be installed in 9 locations in the southwest neighborhood	27	1 duplicate sample per 20 samples	Air Toxics QA/QC methods are presented in the Laboratory Quality Assurance Program (NELAP Quality Manual, Rev. 21.2 date 8/09) and TO-14/TO-15 - Volatile Organic Compounds Methods Manual Section 7 (Rev. 17.1, date 12/09).
Soil-gas sampling points will be installed in 3 locations in the east neighborhood	9	1 duplicate sample per 20 samples	
<b>Description of Groundwater Samples</b>			
Groundwater samples will be collected from permanent monitoring wells GM-25 and GM-77S	2	1 duplicate sample per 20 samples 1 equipment blank <sup>(1)</sup> per 20 samples 1 trip blank per cooler	TestAmerica QA/QC methods are presented in the Quality Assurance Manual (Document No. NC-QAM-001, Rev. 0, effective date 1/1/2008); Corporate Quality Management Plan (Document No. CA-Q-M-002, Rev. 0, effective date 1/12/09); and Determination of Volatile Organics by GC/MS Based on Methods 8260B and 8260A (North Canton SOP No. NC-MS-019, Rev. 1, effective date 1/7/09).
Groundwater table samples will be collected in the same soil boring as the soil-gas point in the southwest neighborhood	9	1 duplicate sample per 20 samples 1 equipment blank <sup>(1)</sup> per 20 samples 1 trip blank per cooler	
Groundwater table samples will be collected in the same soil boring as the soil-gas point in the east neighborhood	3	1 duplicate sample per 20 samples 1 equipment blank <sup>(1)</sup> per 20 samples 1 trip blank per cooler	

Target compounds and analytical methods are presented on Table B-1.

QA/QC - Quality Assurance/Quality Control

(1) Equipment blank will be collected on groundwater sampling equipment that is nondedicated.

(2) Laboratory QC procedures referenced in this table are presented in Appendix B.

ARCADIS

**Attachment 1**

**DLZ Ohio, Inc.**

Consolidated Soil Indexing  
Formulas, 1997

Quality Manual, 2008

# **Civil Engineering Reference Manual for the PE Exam**

**Sixth Edition**

**Michael R. Lindeburg, PE**

## In the ENGINEERING LICENSING EXAM AND REFERENCE SERIES

Engineer-In-Training Reference Manual  
EIT Review Manual  
Engineering Fundamentals Quick Reference Cards  
Engineer-In-Training Sample Examinations  
Mini-Exams for the E-I-T Exam  
1001 Solved Engineering Fundamentals Problems  
Fundamentals of Engineering Exam Study Guide  
Diagnostic FE Exam for the Macintosh  
Fundamentals of Engineering Video Series:  
Thermodynamics

### Civil Engineering Reference Manual Quick Reference for the

Civil Engineering PE Exam  
Civil Engineering Sample Examination  
Civil Engineering Review Course on Cassettes  
101 Solved Civil Engineering Problems  
Seismic Design of Building Structures  
Seismic Design Fast  
345 Solved Seismic Design Problems  
Timber Design for the Civil and Structural  
Professional Engineering Exams  
246 Solved Structural Engineering Problems  
**Mechanical Engineering Reference Manual**  
Mechanical Engineering Quick Reference Cards  
Mechanical Engineering Sample Examination  
101 Solved Mechanical Engineering Problems  
Mechanical Engineering Review Course  
on Cassettes  
Consolidated Gas Dynamics Tables  
Fire and Explosion Protection Systems

### Electrical Engineering Reference Manual Quick Reference for the

Electrical Engineering PE Exam  
Electrical Engineering Sample Examination  
**Chemical Engineering Reference Manual  
for the PE Exam**  
Quick Reference for the  
Chemical Engineering PE Exam  
Chemical Engineering Practice Exam Set  
**Land Surveyor Reference Manual**  
Land Surveyor-In-Training Sample Examination  
1001 Solved Surveying Fundamentals Problems

## In the GENERAL ENGINEERING and CAREER ADVANCEMENT SERIES

How to Become a Professional Engineer  
Getting Started as a Consulting Engineer  
The Expert Witness Handbook:  
A Guide for Engineers  
Engineering Your Job Search  
Engineering Your Start-Up  
Engineering Your Writing Success  
Intellectual Property Protection:  
A Guide for Engineers  
High-Technology Degree Alternatives  
Metric in Minutes  
Engineering Economic Analysis  
Engineering Law, Design Liability, and  
Professional Ethics  
Engineering Unit Conversions  
Calculus Refresher for the  
Fundamentals of Engineering Exam

## CIVIL ENGINEERING REFERENCE MANUAL Sixth Edition

Copyright © 1997 by Professional Publications, Inc. All rights reserved. No part of this publication may be reproduced, stored in a retrieval system, or transmitted, in any form or by any means, electronic, mechanical, photocopying, recording, or otherwise, without the prior written permission of the publisher.

Printed in the United States of America

Professional Publications, Inc.  
1250 Fifth Avenue, Belmont, CA 94002  
(415) 593-9119  
www.ppi2pass.com

Current printing of this edition: 5

### Library of Congress Cataloging-in-Publication Data

Lindeburg, Michael R.

Civil engineering reference manual for the PE exam / Michael R.  
Lindeburg. -- 6th ed.

p. cm.

Rev. ed. of: Civil engineering reference manual.

Includes index.

ISBN 1-888577-03-7 (hardcover)

1. Civil engineering--United States--Examinations--Study guides.
2. Civil engineering--Handbooks, manuals, etc. I. Lindeburg, Michael R. Civil engineering reference manual. II. Title.

TA159.L54 1996

624'.076--dc20

96-44245

CIP

# SOILS

## Consolidated Soil Indexing Formulas (Specific Gravity = G)

	property	saturated sample ( $W_s, W_w, G,$ are known)	unsaturated sample ( $W_s, W_w, G, V$ are known)	supplementary formulas relating measured and computed factors			
volume components	$V_s$ volume of solids		$\frac{W_s}{G\rho_w}$	$V - (V_a + V_w)$	$V(1 - n)$	$\frac{V}{1 + e}$	$\frac{V_v}{e}$
	$V_w$ volume of water		$\frac{W_w}{\rho_w^*}$	$V_v - V_a$	$SV_v$	$\frac{SV_v e}{1 + e}$	$SV_s e$
	$V_a$ volume of air or gas	zero	$V - (V_s + V_w)$	$V_v - V_w$	$(1 - S)V_v$	$\frac{(1 - S)V_v e}{1 + e}$	$(1 - S)V_s e$
	$V_v$ volume of voids	$\frac{W_w}{\rho_w^*}$	$V - \frac{W_s}{G\rho_w}$	$V - V_s$	$\frac{V_s n}{1 - n}$	$\frac{V_v e}{1 + e}$	$V_s e$
	$V$ total volume of sample	$V_s + V_w$	measured	$V_s + V_a + V_w$	$\frac{V_s}{1 - n}$	$V_s(1 + e)$	$\frac{V_v(1 + e)}{e}$
	$n$ porosity		$\frac{V_v}{V}$	$1 - \frac{V_s}{V}$	$1 - \frac{W_s}{GV\rho_w}$	$\frac{e}{1 + e}$	
	$e$ void ratio		$\frac{V_v}{V_s}$	$\frac{V}{V_s} - 1$	$\frac{GV\rho_w}{W_s} - 1$	$\frac{W_w G}{W_s S}$	$\frac{n}{1 - n} \left  \frac{wG}{S} \right.$
weights for specific sample	$W_s$ weight of solids		measured	$\frac{W_s}{1 + w}$	$GV\rho_w(1 - n)$	$\frac{W_w G}{eS}$	
	$W_w$ weight of water		measured	$wW_s$	$S\rho_w V_v$	$\frac{eW_s S}{G}$	$V \cdot \rho_D \cdot w$
	$W_t$ total weight of sample		$W_s + W_w$	$W_s(1 + w)$			
weights for sample of unit volume	$\rho_D$ dry unit weight	$\frac{W_s}{V_s + V_w}$	$\frac{W_s}{V}$	$\frac{W_t}{V(1 + w)}$	$\frac{G\rho_w}{1 + e}$	$\frac{G\rho_w}{1 + wG/S}$	
	$\rho_T$ wet unit weight	$\frac{W_s + W_w}{V_s + V_w}$	$\frac{W_s + W_w}{V}$	$\frac{W_t}{V}$	$\frac{(G + Se)\rho_w}{1 + e}$	$\frac{(1 + w)\rho_w}{w/S + 1/G}$	$\rho_D(1 + w)$
	$\rho_{SAT}$ saturated unit weight	$\frac{W_s + W_w}{V_s + V_w}$	$\frac{W_s + V_v\rho_w}{V}$	$\frac{W_s}{V} + \left(\frac{e}{1 + e}\right)\rho_w$	$\frac{(G + e)\rho_w}{1 + e}$	$\frac{(1 + w)\rho_w}{w + 1/G}$	
	$\rho_{SUB}$ submerged (buoyant) unit weight		$\rho_{SAT} - \rho_w^*$	$\frac{W_s}{V} - \left(\frac{1}{1 + e}\right)\rho_w^*$	$\left(\frac{G + e}{1 + e} - 1\right)\rho_w^*$	$\left(\frac{1 - 1/G}{w + 1/G}\right)\rho_w^*$	
combined relations	$w$ moisture content		$\frac{W_w}{W_s}$	$\frac{W_t}{W_s} - 1$	$\frac{Se}{G}$	$S \left[ \frac{\rho_w^*}{\rho_D} - \frac{1}{G} \right]$	
	$S$ degree of saturation	1.00	$\frac{V_w}{V_v}$	$\frac{W_w}{V_v\rho_w^*}$	$\frac{wG}{e}$	$\frac{w}{\frac{\rho_w^*}{\rho_D} - \frac{1}{G}}$	
	$G$ specific gravity		$\frac{W_s}{V_s\rho_w}$	$\frac{Se}{w}$			

$\rho_w$  is the density of water. Where noted with an asterisk (\*) use the actual density of water. In other cases, use 62.4 lbm/ft<sup>3</sup>.

**CONSTRUCTION MATERIALS LABORATORY AND  
CONSTRUCTION MONITORING AND TESTING SERVICES  
QUALITY ASSURANCE/QUALITY CONTROL MANUAL**

**CONTENTS**

**PAGE**

INTRODUCTION.....2

STATEMENT OF FIRM’S OVERALL OPERATING PHILOSOPHY .....4

MEMBERSHIP IN TECHNICAL AND PROFESSIONAL SOCIETIES .....6

CLIENTELE.....8

ORGANIZATION AND ORGANIZATIONAL POLICIES:

Name of Firm, Address of Main Office, Telephone Number ..... 10

Name of Firm, Address of Construction Materials Laboratory, Telephone Number..... 10

Name of Responsible Firm Member and Project Management Members ..... 10

STAFF AND FACILITIES:

Construction Materials Laboratory Facilities ..... 13

Construction Materials Laboratory Position Descriptions ..... 13

Construction Materials Laboratory Training Procedures..... 13

Construction Materials Laboratory Internal Quality Control Program..... 14

Construction Monitoring and Testing Services Quality Control Program ..... 15

Management Review..... 15

Radiation Safety..... 15

Construction Monitoring and Testing Services Training Procedures ..... 16

Laboratory Safety..... 17

EQUIPMENT:

Geotechnical Testing Services Inventory List..... 19

Laboratory Equipment Calibration and Verification.....20

In-House Calibration and Verification Procedures.....20

Field Equipment Calibration and Verification.....21

CONSTRUCTION MATERIALS LABORATORY TESTING SERVICES .....23

CONSTRUCTION MONITORING AND TESTING SERVICES .....26

**CONTENTS – CONT.**

**PAGE**

TEST RECORDS AND REPORTS:

Construction Materials Laboratory Testing Procedures .....	29
Typical Testing Report Forms .....	30

SAMPLE MANAGEMENT .....	32
-------------------------	----

DIAGNOSTIC AND CORRECTIVE ACTION:

Inspection Programs .....	34
Procedures for Correcting Poor Testing Results.....	34
External Technical Complaints.....	36
Subcontracting.....	36

CONSTRUCTION MATERIALS LABORATORY QUALITY SYSTEM:

Internal Quality System Review .....	38
Construction Material Laboratory Accreditation's.....	38

**APPENDIX I**

Division Organizational Chart

**APPENDIX II**

Construction Materials Laboratory Floor Plan

**APPENDIX III**

Position Descriptions

**APPENDIX IV**

Resumes for Barry Wong  
Jamie North  
Eugene Sabic

**APPENDIX V**  
Master Task Sign-Off Sheet

**CONTENTS – CONT.**

**APPENDIX VI**

Equipment

**APPENDIX VII**

In-House Calibration Procedures:

Procedure #1 Ovens  
Procedure #2 Vacuum System  
Procedure #3 Manual Hammers  
Procedure #4 Conical Mold, Tamper  
Procedure #5 Straightedges  
Procedure #6 Grooving Tool  
Procedure #7 Liquid Limit Device  
Procedure #8 Hydrometer Bulbs  
Procedure #9 California Bearing Ratio Equipment  
Procedure #10 Sodium Sulfate Containers  
Procedure #11 Compaction Molds  
Procedure #12 Mechanical Sieve Shakers  
Procedure #13 One-Dimensional Consolidation Load Indications  
Procedure #14 Unit Weight Measure  
Procedure #15 Wire Cloth Sieves  
Procedure #16 Laboratory Moisture Cans  
Procedure #17 Digital Calipers  
Procedure #18 Mechanical vs. Manual Marshall Hammers  
Procedure #19 NTO Burn-off Oven & Aggregate Correction Factor  
Procedure #20 In-house Verification of Dial Indicators  
Procedure #21 Marshall Molds  
Procedure #22 Thermometer Accuracy  
Procedure #23 Marshall Hammers  
Procedure #24 Pressure Meters

**APPENDIX VIII**

Typical Test Report Forms

**APPENDIX IX**

Approved Subcontractor List

**APPENDIX X**

Revision Logs

# **INTRODUCTION**



## ***Introduction***

DLZ, Ohio is a professional engineering firm serving Ohio and the surrounding states with a staff of experienced engineers, geologists, environmental specialists, technicians, drillers, and support personnel. DLZ maintains its own well-equipped soils and materials laboratory, field-testing equipment, drill rigs, and support equipment.

Barry Wong is in charge of field and laboratory testing and is responsible for the field Quality Assurance/Quality Control. Jamie North assists in the supervision and training of field and laboratory personnel and serves as the Assistant Construction Field Services Supervisor. Eugene Sabic and Jamie North supervise the laboratory testing, are in charge of laboratory training, and are the laboratory Quality Assurance/Quality Control Coordinators.

DLZ adheres to the guidelines presented in The American Association of State Highway and Transportation Officials (AASHTO) Specification R-18 for establishing and maintaining a Quality System for its Construction Materials Testing Laboratory. In addition, the DLZ Construction Materials Laboratory (CML) is certified and accredited by the AASHTO Materials Reference Laboratory (AMRL) and the Cement and Concrete Reference Laboratory (CCRL). The DLZ CML also adheres to the further requirements set forth by The American Society for Testing and Materials (ASTM) Specifications D-3666 (Requirements for Agencies Engaged in Testing of Bituminous Paving Materials), D-3740 (Requirements for Agencies Engaged in Soil and Rock Testing) and C1077 (Requirements for Agencies Engaged in testing Concrete and Concrete Aggregates). We are AMRL certified since 1998.

DLZ and its predecessor companies have been in business in Columbus since 1968. The Company has built and maintained a sound reputation by providing competent and efficient engineering, testing, and related services to engineers, architects, contractors, public agencies, and owners in Ohio and surrounding states. Today, DLZ has a staff of over 250 experienced personnel.

**STATEMENT OF FIRM'S OVERALL OPERATING PHILOSOPHY  
AND ORGANIZATIONAL CHARACTERISTICS**

***Statement of Firms Overall Operating Philosophy and Organizational Characteristics***

DLZ is committed to achieving strong growth as a company while at the same time providing architectural, engineering, and environmental services to the complete satisfaction of each of our clients. The management at DLZ is committed to operating the firm in a professional and cost conscience manner while not loosing sight of the company's larger mission: To build the Midwest's leading full-service architectural, engineering, and environmental firm. The formula for success at DLZ is simple: Fully satisfy each our clients, manage our costs, capitalize on our strengths and our position in the market, and seize every opportunity for growth and success.

DLZ feels that an effective combination of management and technical support is essential for the success of each of our projects. Based on this reasoning, every contract at DLZ has the full support of DLZ's top management, thereby ensuring responsible and efficient planning and implementation. In addition, our Project Managers have access to all the resources and skills of the firm. This philosophy is the key to the complete satisfaction of our clients as well as to the success of our company.

**MEMBERSHIP IN TECHNICAL AND PROFESSIONAL SOCIETIES**



### ***Membership in Technical and Professional Societies***

In order to stay in touch with new ideas and technologies, DLZ maintains membership in various technical and professional societies such as:

- American Concrete Institute
- American Society of Civil Engineers
- American Society for Testing and Materials
- Institute of Civil Engineers
- National Society of Professional Engineers
- National Ground Water Association
- Ohio Society of Professional Engineers
- Ohio Ready Mixed Concrete Association
- National Institute for Certification in Engineering Technologies
- American Association of State Highway and Transportation Officials - Materials Reference Laboratory
- Flexible Pavement of Ohio

**CLIENTELE**

### *Clientele*

DLZ has worked with a variety of groups and interests such as:

- U.S. Army Corps of Engineers
- Landfill operators/owners
- Design architects and engineers
- Federal, state, county, and municipal government agencies
- Commercial and industrial developers
- Design-constructors, contractors, and builders
- Church organizations, fraternal groups, and civic groups
- School systems and universities
- Various industries
- Recreational facilities developers
- Private home owners
- Material producer
- Quarrying and mining interests



## **ORGANIZATION AND ORGANIZATIONAL POLICIES**

***Name of Firm, Address of Main Office, Telephone Number:***

DLZ Ohio, Inc.  
6121 Huntley Road  
Columbus, Ohio 43229  
Phone: 614/848-4141  
Fax: 614/848-6712

***Name of Firm, Address of Construction Materials Laboratory, Telephone Number:***

DLZ Ohio, Inc.  
6121 Huntley Road  
Columbus, Ohio 43229  
Phone: 614/888-0576  
Fax: 614/888-6415

DLZ Ohio, Inc. a subsidiary of the DLZ Corporation, was established in 1995 by the consolidation of Dodson-Lindblom Associates, Inc. (including Mason - de Verteuil Geotechnical Services), Stilson & Associates, Inc., and JDJ&A, Inc. With this consolidation, DLZ is capable of providing top quality engineering and architectural services within schedule constraints for each project.

***Name of Responsible Firm Member and Project Management Members***

**Mr. A. James Siebert, P.E.**, Senior Vice President, will serve as **Project Principal** and provide corporate level commitment and management review. During the course of his career he has been responsible for numerous state, federal, and local renovation projects. He is committed to performing all work in a timely manner and to the complete satisfaction of each client. Mr. Siebert recently served as Project Principal on projects completed for the U.S. Army Corps of Engineers, the City of Columbus, the Greater Cleveland Regional Transit Authority, the Ohio Department of Transportation, and others.

**Mr. David Cutlip**, Vice President, will provide additional corporate level commitment and management review. As Vice President of DLZ, Mr. Cutlip is active in overseeing the day-to-day operations of DLZ's technical support staff. Mr. Cutlip has extensive experience in construction and cost estimating.

**Mr. Arthur E. Nix, P.E.**, Geotechnical Engineering Division Manager. Mr. Nix is experienced in all aspects of geotechnical and subsurface investigations including drilling, sampling, field and laboratory testing, site evaluation, and preparation of reports. His project experience includes slope stability analyses,

foundation investigations for floodwalls and dams, and geotechnical investigations and recommendations for airport and roadway projects.

**Mr. Barry Wong, P.E.**, Construction Services Division Manager will be responsible for ensuring field and laboratory **Quality Assurance and Quality Control**. Mr. Wong has been involved in many geotechnical and construction related projects including subsurface investigation, laboratory testing, roadway pavement designs, field observation and evaluation, and foundation investigations.

**Mr. Jamie North** will serve as the **Construction Field Services Supervisor** and will assist in providing Laboratory Quality Assurance and Quality Control. Mr. North has extensive experience in many aspects of laboratory testing and construction-related observation.

**Mr. Eugene J. Sabic** will serve as the **Construction Materials Laboratory Manager** and will provide **Laboratory Quality Assurance and Quality Control**. Mr. Sabic has extensive experience as a Senior Technician with DLZ and has been responsible for a wide variety of laboratory tests for soils, aggregates, concrete, and asphalt. Mr. Sabic's field and laboratory experience allows him to handle a variety of construction related conditions.

The complete organizational structure of DLZ (including the Construction Materials Laboratory) is presented on the organizational charts found in Appendix I.

## **STAFF AND FACILITIES**

### ***Construction Materials Laboratory Facilities***

The DLZ CML is located at 6121 Huntley Road in Columbus, Ohio. The total laboratory testing area is over 2000 square feet. A floor plan of DLZ CML is presented in Appendix II.

### ***Construction Materials Laboratory – Position Description***

The descriptions for CML positions are presented in Appendix III. In addition, the resumes for technical staff are presented in Appendix IV.

### ***Construction Materials Laboratory Training Procedures***

As part of the DLZ internal Quality Control Program, all Construction Materials Laboratory personnel are trained to ensure that all standard test methods and procedures are properly followed. CML personnel are initially trained by the laboratory supervisor, field supervisory personnel, or a Laboratory Geotechnician Level II or higher as to the standard testing methods and procedures. Technicians are required to read and be familiar with the appropriate ASTM, AASHTO, ACI, EPA, and/or U.S. Army Corps of Engineers procedures on all tests they perform.

During the training period the instructor doing the training first explains and demonstrates the test. The trainee then performs the test under supervision until successful, whereupon the instructor witnesses the successful completion of the test and signs off on Task Sign Off Sheet. After a trial period with intermittent checks, the trainee is signed off on a Master Task Sign Off Sheet and considered competent to perform that test. The final sign off on the Master Task Sign Off Sheet is done only by the CML Supervisor or Manager when he or she has observed the technician performing the task without assistance for an extended period of time. The Manager and Laboratory Supervisor are responsible for the CML training program, competency evaluations, and maintenance of all training records.

Copies of all training records (including all Individual Task Sign Off Sheets and Master Task Sign Off Sheets) are given to the Division Manager for review. The originals of these training records are posted on a specific wall in the CML for review and comments by all DLZ employees.

Experienced technicians are supported and encouraged to observe and reinstruct other staff members when a deviation in procedure or results is observed. In addition, any technician returning to the CML after an extended absence must be formally reinstructed and must again demonstrate competence in each test which the technician is to perform.

Samples of the Individual Task Sign Off Sheet and Master Task Sign Off Sheet for the DLZ CML are presented in Appendix V.

### ***Construction Materials Laboratory Internal Quality Control Program***

The purpose of the Construction Materials Laboratory Quality Control Program at DLZ is to insure that all tests are performed by trained and competent technicians in accordance with recognized standard procedures and using properly calibrated equipment. The Quality Control Program attempts to insure accuracy in testing results by checking for errors in execution of the test procedure and computation of the test results and correcting any errors which are detected. The Quality Control Program also attempts to prevent any reoccurrence of error by re-instructing the appropriate laboratory personnel when deviations occur in either the test procedure or computation of results.

All geotechnical laboratory testing at DLZ is performed in accordance with the appropriate ASTM, AASHTO, ACI, EPA, and/or U.S. Army Corps of Engineers Standards. The CML at DLZ maintains an updated library of these references.

In order to ensure Quality Control, the CML at DLZ participates in the U.S. Army Corps of Engineers Inspection Program wherein laboratory personnel, procedures, and equipment are periodically inspected by The U.S. Army Corps of Engineers. In addition, the CML at DLZ adheres to the AMRL proficiency program for certain soils and aggregate tests and also participates in the AMRL and CCRL Inspection Program.

Internal Laboratory testing (internal auditing) is required of laboratory technicians as part of a yearly performance evaluation program to insure continued competency, which is observed, documented and records kept by the CML Managers. Each technician is evaluated for competency for each test that they will be responsible for performing (a list of all possible tests can be found in Appendix V). At a minimum, each employee whom work in the laboratory are evaluated for competency in the following areas:

Atterberg limits, Grain Size Analysis and Water Content: Once a year each CML technician performs these in-house samples. The technicians are judged on accuracy, consistency, and consensus.

Proctor curves: Once a year all CML technicians are required to generate a Proctor curve on the same in-house standard material. The results of all technicians are then compared for accuracy, consistency, and consensus. This includes adhering to the specification set forth in AASHTO T99

Section 14.2 which requires that multi-operator results on the same material must differ by no more than 15 percent of their mean for optimum moisture and 4.5 lbs/ft<sup>3</sup> for maximum dry density.

All CML technicians are subject to continuous review and reinstruction by the CML Managers or Laboratory Geotechnician Level II or higher. When a deviation in procedure or results is observed, the technician responsible for the deviation is formally reinstructed and must again demonstrate competence in the relevant testing procedure. Any such actions will be documented and records will be kept by the CML Managers.

### ***Construction Monitoring and Testing Services Quality Control Program***

The purpose of the Construction Monitoring and Testing Services Quality Control Program at DLZ is to insure that all field-testing is performed by trained and competent technicians in accordance with recognized standard procedures and using properly calibrated equipment. The Quality Control Program attempts to insure accuracy in the testing results by checking for errors in execution of the test procedure and computation of the test results and correcting any errors which are detected. The Quality Control Program also attempts to prevent any reoccurrence of error by reinstructing the appropriate field personnel when deviations occur in either the test procedures or computation of results.

All field testing at DLZ is performed in accordance with the appropriate ASTM, ACI, AASHTO, EPA, and/or U.S. Army Corps of Engineers Standards. DLZ maintains an updated library of these references.

The Construction Monitoring and Testing Services division at DLZ participates in the U.S. Army Corps of Engineers Inspection Program where certain field testing and inspection equipment is periodically inspected by U.S. Army Corps Personnel.

### ***Management Review***

During the first quarter of each year, or whenever it is deemed necessary, the DLZ Construction Services Managers will meet with the DLZ Vice President(s) to have a Management Review, to review the previous year's actions. Records shall be kept on the Management Review form to insure that all necessary topics are discussed; Audits and Accreditations, Personnel, Proficiency Testing, Purchasing, Feedback and Recommendations for Improvement.

## ***Radiation Safety***

Prior to using or transporting a nuclear moisture-density gauge, all technicians as well as field supervisory personnel are required to attend a Nuclear Regulatory Commission (NRC) recognized Radiation Safety Class. Only upon successful completion of this class are field personnel permitted to independently use or transport a nuclear gauge.

All technicians and field personnel are required to keep their assigned radiation film badges on them when using or transporting a nuclear gauge. These film badges are returned each month and checked for possible exposure to radioactive material.

During transportation of a nuclear gauge, all field personnel are required to maintain a copy of the Bill of Lading of Radioactive Material in the nuclear gauge transportation box as well a copy in the transporting vehicle. The Bill of Lading contains the following information:

1. Name and address of company transporting the nuclear gauge.
2. Type and quantities of radioactive material presents.
3. Emergency response phone numbers.
4. List of potential hazards involved with the radioactive materials.
5. List of emergency actions to be taken if nuclear sources are breached.

During any transportation of a nuclear gauge, or when a gauge is in the field but not being used, all technicians are required to keep the nuclear gauge locked and secured in the appropriate transportation box and stored in the portion of their vehicle furthest from the driver or any passengers. This involves either locking the gauge in the trunk of a car or chaining and locking the gauge in the bed of a truck. When nuclear gauges are in the field but not being used, the gauges are kept locked to ensure that the radioactive sources are not accidentally exposed. When not in the field, all nuclear gauges are required to be stored in a secured area on the premises of the DLZ Construction Materials Laboratory.

## ***Construction Monitoring and Testing Services Training Procedures***

As part of the DLZ internal Quality Control Program, all field personnel are trained in order to ensure that all standard test methods and procedures are properly followed. All field technicians are initially trained in the laboratory by the laboratory supervisor, field supervisor, or senior technician as to the standard field testing methods and procedures. Technicians are required to read and be familiar with this manual as well as the appropriate ASTM, AASHTO, ACI, EPA, and/or U.S. Army Corps of Engineers procedures on all tests they perform.

During the in-house training period, the instructor doing the training first explains and demonstrates the test. The trainee then performs the test under supervision until successful, where upon the instructor witnesses the successful completion of the test and signs off on a task sheet. After a trial period with intermittent checks, the trainee is signed off on a master sheet for each test and considered competent to perform the test.

After the in-house training period, the technician is introduced to the field for a period of field training. This period usually entails 7 to 10 days of on-site training with either the field supervisor or a senior technician. After the 7 to 10 day field training period the technician is assigned to a job site with intermittent, regular on-site checks by the field supervisor.

All technicians are subject to regular, on-site checks by the field supervisor. During these checks the field supervisor will inspect the technicians paperwork, testing methods, calculations, and general job-site procedures. Any deviation in these areas results in reinstruction by the field supervisor.

For concrete testing, all technicians are initially trained in-house and then in the field by either the field supervisor or a senior technician. All technicians are encouraged to become certified by the American Concrete Institute (ACI).

Experienced technicians are supported and encouraged to observe and reinstruct other staff members when a deviation in procedure is observed. All technicians returning to the field after an extended absence are formally reinstructed and must again demonstrate competence in each test.

The construction monitoring and testing services division at DLZ maintains and uses cell phones. These communication devices are used to ensure that all field technicians are capable of contacting the field supervisor whenever necessary.

All field technicians are subject to continuous review and reinstruction by the CML Supervisor, field supervisory personnel, or Laboratory Geotechnician Level II or higher. When a deviation in procedure or results is observed, the technician responsible for the deviation is formally reinstructed and must again demonstrate competence in the relevant testing procedure.

### ***Laboratory Safety***

As part of all standard laboratory practices, good personal hygiene is maintained by all employees while working in the laboratory. These practices include washing exposed body parts after handling samples and

wearing appropriate clothing at all times. In addition, if potentially contaminated samples are to be handled, gloves, respiratory masks, and/or safety glasses are worn.

If potentially contaminated samples are to be handled in the laboratory, information regarding the potential hazards along with any special handling requirements must accompany the sample at all times. This information is made available by the Project Manager and/or the Health and Safety Officer. All special handling procedures for specific chemicals or samples contaminated with these chemicals are performed in accordance with the recommendations provided in the chemical specific Material Safety Data Sheets (MSDS). If the MSDS are not readily available for such chemicals, the Health and Safety Officer is immediately notified. The Health and Safety Officer is responsible for obtaining the relevant MSDS or other specific hazard information for that chemical.

## **EQUIPMENT**

***Geotechnical Testing Services - Inventory List***

An essential equipment list associated with and used by the DLZ Geotechnical Testing Services Department is as follows:

<b>Lab Equipments</b>
15 Triaxial / Permeability Chambers
Triaxial Load Frame
6 Soil Consolidometers
CBR Equipment
Soil Mixers
Mechanical Soil Compactor
Direct Shear
Laboratory Sieve Sifter
Soil Compaction Molds
Asphalt Content Gage
NCAT Burn-off Oven
Asphalt Rice Test
Asphalt Centrifuge Extraction
Forney Concrete Compression Machine
Concrete Saw
De-airing Water Tank
Rock Point Load
Cured-in-Place Pipeline Testing Equipment

DLZ has purchased the computer software GAGEtrak from Cyber Metrics to organize all equipment used for the CML Laboratory.

### ***Laboratory Equipment Calibration and Verification***

DLZ ensures that the Construction Materials Laboratory test equipment presented in Appendix VI is calibrated or verified according to the specified requirements and at intervals not to exceed the maximum calibration interval shown. All calibration and verification records are kept in the room that the corresponding test is performed. All certificates and other documents that establish the traceability of in-house calibration and verification equipment are kept in these binders as well. The Department Manager and the CML Supervisor are responsible for the maintenance of all equipment calibration and verification records.

The Manager and the CML Supervisor are responsible for ensuring that all appropriate test equipment is calibrated or verified accordingly. At the beginning of each quarter, the CML Supervisor and Manager will refer to the Quality Manual and calibration records in order to determine which test equipment is to be calibrated or verified during that quarter. The CML Supervisor and Manager will then instruct the appropriate laboratory personnel or outside contractor to perform the necessary calibration or verification and will also indicate the date by which the calibration or verification is to be completed. These personnel will fill out the appropriate calibration or verification record forms and return them to the CML Supervisor when the calibration or verification of a particular piece of equipment is completed.

Newly acquired testing equipment is visually inspected for defects and then calibrated or verified before being placed into service. If the newly acquired equipment comes with appropriate documentation indicating that it has already been calibrated or verified by the manufacturer according to the appropriate standards, the equipment is still verified in-house in order to ensure the accuracy of the calibration.

Out of calibration equipment or equipment which is defective is removed from service as soon as the unacceptable condition is recognized. The equipment is tagged Defective - Do Not Use or Out of Calibration - Do Not Use and set aside in a discernible portion of laboratory. The equipment is then calibrated or repaired prior to being returned to service. The Manager is given copies of all calibration and verification records on a monthly basis. The Manager and CML Supervisor are also verbally notified immediately whenever any equipment has been found to be defective or out of calibration.

### ***In-house Calibration and Verification Procedures***

In addition to the applicable ASTM and/or AASHTO Standards, The Construction Materials Laboratory adheres to the procedures established by DLZ when conducting in-house calibration and verification of certain laboratory testing equipment. In-house procedures presented in Appendix VII.

### ***Field Equipment Calibration and Verification***

Calibration and Maintenance of construction monitoring and testing services equipment is performed on a regular basis. All Troxler Nuclear Gauges are periodically shipped to Troxler, Inc. for calibration and maintenance. All other equipment is calibrated in-house according to the U.S. Army Corps of Engineers Calibration Methods and/or ASTM. Specifically:

Proctor mold volumes are redetermined every two years, closely approximately the 1000 use period required by ASTM.

The weight (tare) of the Proctor mold is redetermined each time the mold is used.

Sample cans are reweighed yearly.

The volume of the concrete unit weight container is redetermined each time it is used.

The calibration factor for each pressure method concrete air meter is determined each time the meter is maintained, but at least once a year.

All Troxler Nuclear Gauges are subjected to a stat and drift test once a year, or when test results are questionable. All Troxler Nuclear Gauges are standardized at least once a day when the gauge is to be used.

Several representative soil samples are obtained in the field on a daily basis and returned to the laboratory for moisture content determinations. These moisture contents are then used to check the accuracy of the nuclear moisture-density gauges.

All field scales are checked for accuracy at least once a year, or whenever results are questionable.

All radiation film badges are exchanged each month and checked for exposure.

All Troxler Nuclear Gauges are wipe tested every six month to ensure that the radioactive material sources have not been compromised.

All field lock levels are adjusted at least once a year, or whenever their accuracy is suspect.

**CONSTRUCTION MATERIALS LABORATORY  
TESTING SERVICES**

## ***Construction Materials Laboratory Testing Services***

DLZ provides a full range of testing services related to construction materials, including those standard tests which are listed below. DLZ frequently handles requests for additional non-standard testing procedures. Non standard tests are often performed on an as needed basis.

### **SOIL**

- Permeability Testing, flexible wall (19 cells - 1.4" to 4.0" diameter)
- Permeability of Granular Material, Constant Head (fixed wall)
- Triaxial Compression Testing (9 cells)
- Consolidation Testing (4 load frames, up to 32 tsf)
- Direct Shear (2.5" diameter)
- Sieve and Hydrometer Analysis (Grain-Size Analysis)
- Moisture-Density Relationship (Standard and Modified Proctors)
- Atterberg Limits
- Shrinkage Limit
- Specific Gravity
- Organic Content (Loss On Ignition)
- Relative Density (0.1 and 0.5 ft<sup>3</sup>)
- Unconfined Compression Testing
- California Bearing Ratio (CBR)
- Corrosion Potential of Soils (Electrical Resistivity Method)

### **ROCK**

- Unconfined Compressive Strength (Peak and Stress-Strain)
- Slake Durability Index
- Total Hardness Testing
- Hydraulic Conductivity
- Direct Shear Testing
- Pullout Testing

### **AGGREGATE**

- Sodium Sulfate Soundness
- Sieve Analysis (Grain-Size Analysis)
- Specific Gravity and Absorption
- Percentage of Deleterious Materials
- Insoluble Residue Test

Percent Crushed Particles

**PORTLAND CEMENT CONCRETE**

Compressive Strength of concrete cylinders  
Flexural Strength of concrete beams  
Chloride Ion Testing & Carbonation Testing (Phenolphthalein Testing)

**MASONRY**

Compressive Strength of mortar and grout cubes  
Compressive Strength of brick and block  
Unit Weight, Absorption, etc.

**ASPHALT CONCRETE**

Extraction and Gradation  
Theoretical Maximum Specific Gravity (Rice Method)  
Bulk Specific Gravity  
Marshall Pill  
Stability and Flow

**OTHER**

Fire Proofing Materials Testing  
Roofing Materials Testing  
Rock Core/Reinforcing Steel Pull-Out Test (Anchor Test)

**CONSTRUCTION MONITORING AND TESTING SERVICES**

## ***Construction Monitoring and Testing Services***

DLZ is staffed and well equipped to perform field inspection and testing services for soils and foundations, Portland cement concrete, asphaltic concrete, structural steel, roofing, and other construction materials.

### **SOILS AND FOUNDATIONS**

- Field Moisture-Density Testing (both nuclear densometer and sand cone methods)
- One-Point Proctor Determination
- Field Permeability Testing (Boutwell Permeameters)
- Monitoring of engineered fills
- Monitoring of footing, pile, and caisson installation
- Pile Load Testing
- Field California Bearing Ratio (CBR)
- Plate Load Testing
- Percolation Testing
- Corrosion Potential of Soils (electrical resistivity method)
- Testing of tie-back anchors

### **PORTLAND CEMENT CONCRETE**

- Field Quality Control Testing: Slump, Air Content, Unit Weight, Cylinders, Beams
- Monitoring of concrete placement
- Reinforcing Steel Inspection
- Batch plant inspection
- Coring of existing concrete
- Non-Destructive Testing (Swiss Hammer, Windsor Probe, V-Meter, etc.)
- Evaluation of concrete in service
- Pull-Off Testing and Reinforcing Steel Pull-Out Testing

### **MORTAR AND GROUT**

- Field Monitoring and Quality Control of Masonry Construction
- Mortar Cubes and Grout Cubes for Compressive Strength Testing

### **ASPHALTIC CONCRETE**

- Monitoring of concrete placement
- Batch plant inspection
- Field density and asphalt content testing
- Pavement coring, surveys, and evaluation

Field sampling for laboratory testing

## STRUCTURAL STEEL

- Field erection monitoring
- Bolt torque testing
- Visual weld inspection, stud welding inspection
- Fabrication shop welding inspection

## ROOFING

- New and existing roof inspection and evaluation

## OTHER

- Paint Thickness measurements
- Fire Proofing inspection and testing

## **TEST RECORDS AND REPORTS**

## ***Construction Materials Laboratory Testing Procedures***

All Construction Materials Laboratory (CML) testing at DLZ is performed in accordance with the appropriate ASTM, AASHTO, ACI, EPA, and/or U.S. Army Corps of Engineers Standards. All tests are performed within designated rooms of the CML and the most current appropriate standards and/or specifications for the tests designated to each room are kept in the “Standards and Specifications” binder for that room. An updated copy of the Quality Manual is kept in a separate binder in each room, and all in-house procedures can be found in Appendix VII of the Quality Manual. The CML also maintains a library of other references. During the first quarter of each year, the DLZ managers and supervisors will confirm that the binders have the latest standards and specifications. Old specifications will either be discarded or marked as “Reference Only”, so the most recent standards and specifications are used by DLZ technicians, unless specified.

All laboratory testing is ordered in writing on the appropriate DLZ forms. Each transfer of a sample is recorded on the appropriate forms which are kept with the sample until testing is completed. Laboratory personnel are required to initial and date the appropriate forms for any laboratory testing they were involved with.

When a new laboratory project is begun, the Manager or laboratory supervisor are first responsible for completing the “Schedule of Laboratory Tests” form. This form contains all information relevant to the particular project including:

- 1) Project Name
- 2) Project Number
- 3) Client Name and Contact Person
- 4) Sample(s) Identification
- 5) Standard Test Methods to be used (ASTM, AASHTO, Army Corps, etc.)
- 6) Known deviations from the Standard Test Methods

Once completed, the Schedule of Laboratory Tests form is to be permanently affixed to the inside front cover of the project file folder.

After samples are received in the laboratory, and if requested by the client, they are physically described and classified. The results of the description and classification are recorded on the appropriate DLZ form. The description and classification forms are given to the laboratory supervisor, field supervisory personnel, or the Project Engineer who will check the accuracy of the description and classification.

Requests for laboratory testing are recorded on the "Schedule of Laboratory Tests" form. Each test which is assigned has an independent designated form for that specific test. Once a form has been properly filled out for a given sample, the form remains with the sample until all testing on the sample is completed.

After testing is completed on a sample, the appropriate completed test forms are directed to either the laboratory supervisor, field supervisory personnel, or Project Engineer who will check the accuracy of the testing results and any computations.

Most laboratory test results are entered and computed on an IBM compatible computer using *GeoSystem* software or other software programs specifically developed for the DLZ Construction Materials Laboratory. Occasionally results are reported out in letter or spread sheet format.

If for any reason it is necessary to amend a given test report, the original test report and corresponding test forms are not discarded. These documents are overwritten "error" and placed in the project folder. All amended test reports are prepared only by the Manager, laboratory supervisor, or field supervisory personnel in order to minimize the possibility of additional test report amendments. The Manager and laboratory supervisor are responsible for contacting the client to discuss the test report amendments.

After all laboratory testing for a particular project is completed, the original completed tests forms ("Bench Sheets") are retained on file along with copies of the formal test results given to the Project Engineer or the client. These files are boxed and maintained in an orderly fashion in the basement of the DLZ main office for a period of at least five years. The Manager and laboratory supervisor are responsible for the maintenance and storage of all test records, forms, and reports.

### ***Typical Testing Report Forms***

Several examples of typical test report forms used by the Construction Materials Laboratory at DLZ are presented in Appendix VIII.

## **SAMPLE MANAGEMENT**

## ***Sample Management***

Either the Laboratory Supervisor, the Manager, or field supervisory personnel are responsible for the receipt of all samples from clients. Sample reception includes the maintenance of all appropriate sample documentation as well as inspection of: labeling, shipping methods, packaging types, and condition of containers. Sample reception also includes completion of any chain-of-custody forms as well as checking custody seals, properly filing any relevant forms, supplying verification of receipt of samples, and resolving any problems with samples through the proper chain-of-command.

Laboratory chain-of-custody records for each sample are maintained on the appropriate forms. The custody records generally include the sample identification, the date and time of transfer, and the signatures of both the person relinquishing the samples and the person receiving the samples. These records are not necessarily required for all samples which are delivered to the laboratory unless the samples are suspected of being contaminated. However, a chain-of-custody record might be required from a regulatory or contractual stand-point. The client or the individual at DLZ in charge of a project for which samples are received is responsible for specifying if any chain-of-custody records are to be maintained.

After samples are properly received in the laboratory they are stored in a secured area on the premises of the laboratory. Samples are maintained in an orderly fashion along with all identifying documents. Large bag samples are identified with two index cards containing the project number or identification, sample number and location, and any other necessary identifying information. One card is placed inside of the sample bag while the other is tied or double stapled to the outside of the bag. Bucket and jar samples have this same information permanently affixed to the side of the container or written on the lid in permanent marking.

Hazard information is readily available if samples are suspected of being contaminated. Samples which are suspected of being contaminated are stored in a separate designated area in the laboratory. Access to this area is limited to authorized personnel only. Signs are placed at the entrance of the storage area to document any potential hazards associated with the samples. Precautions are taken to prevent samples from becoming mixed with or contaminated by other samples.

After all laboratory testing is completed the samples are placed into appropriate containers and stored in a secured area for retention for a minimum of six months. The location of each sample being retained is recorded and the location kept on file in the CML. If requested, samples are returned to the client for disposal or additional retention. Otherwise, the samples are properly disposed of under direction of the Laboratory Supervisor or the Manager.

**DIAGNOSTIC AND CORRECTIVE ACTION**

### ***Inspection Programs***

The Construction Materials Laboratory at DLZ participates in the U.S. Army Corps of Engineers Inspection Program wherein laboratory personnel, procedures, and equipment are periodically inspected by the U.S. Army Corps of Engineers. The CML at DLZ also ascribes to the AASHTO Materials Reference Laboratory (AMRL) proficiency program and the Cement and Concrete Reference Laboratory (CCRL) proficiency program for certain soils, aggregate, and concrete tests. The CML at DLZ also participates in the AMRL Inspection Program and is periodically inspected by the Indiana Department of Transportation (INDOT) and several private organizations.

The CML at DLZ uses the results of the AMRL and CCRL proficiency sample testing and on-site inspection programs to identify poor testing procedures or results. Poor testing results are considered any test results beyond two standard deviations from the average values.

### ***Procedures for Correcting Poor Testing Results***

When poor or deficient testing results do occur, the first items to be checked for errors are any mathematical calculations and computer entries. If the cause of the poor testing results is discovered in one of these two areas, it is typically easy to correct. A file is maintained in the DLZ CML which gives examples of various types of mathematical and computer entry errors for laboratory personnel to review.

The CML at DLZ requires that all raw data sheets ("Bench Sheets") be initialed and dated by all laboratory personnel who are involved with a particular test for a given sample. This provides a "paper-trail" which can be further investigated to determine the source of poor or deficient testing results.

If poor or deficient testing procedures or results are identified during the proficiency sample testing or on-site inspections, the following procedures are followed by the DLZ CML in order to correct these deficiencies:

- 1) A meeting is held at the DLZ CML in order to discuss the poor or deficient testing procedures or results. This meeting is attended by the Department Manager, laboratory supervisor, field supervisory personnel, and other relevant DLZ employees.
- 2) The following actions are then initiated in order to correct the poor or deficient testing procedures or results:

Procedures to Follow When Poor Proficiency Sample Results Occur:

- a. Determine if the Agency conducting the proficiency program correctly entered the data submitted to them by the CML.
- b. Determine if the test results obtained were properly transferred to the data sheet submitted to the Agency conducting the proficiency program.
- c. Determine if all calculations leading to the test results obtained were correct.
- d. Determine if the equipment used to perform the tests meets specification requirements.
- e. Determine if the procedures followed when performing the tests conformed to specification requirements.
- f. Take corrective action to repair or replace defective equipment or reinstruct the appropriate laboratory personnel as to the correct testing procedures.

Procedures to Follow When On-Site Inspection Deficiencies Are Reported:

(Apparatus Deficiencies)

- a. Determine if the equipment meets specification requirements.
- b. If the equipment is found to be defective immediately remove the equipment from service and take the necessary steps to repair or replace it.

(Procedural Deficiencies)

- a. Discuss each procedural deficiency with the appropriate personnel and review the proper procedure.
- b. Observe the employee performing the test properly.

(Quality System Deficiencies)

1. The Department Manager shall review each deficiency cited by the evaluator with the responsible employee.
2. Take appropriate action.

If the investigation into the source of poor or deficient testing results reveals nothing, or if it reveals an error which cannot be corrected with the present data, additional sample material is obtained and the test is repeated. The incorrect report and the associated bench sheets are overwritten "error" and placed in the project folder. When a deviation in testing procedures or results does occur, the laboratory personnel responsible for the deviation are formally reinstructed and must again demonstrate competence in the relevant testing procedure. All retraining and reinstruction is recorded on the appropriate Individual Task Sign-Off Sheet.

### ***External Technical Complaints***

Technical complaints from outside of DLZ are treated in essentially the same manner as if the complaint were coming from within DLZ. The company or individual making the complaint is initially directed to either the Department Manager, the Laboratory Supervisor, or field supervisory personnel in order to correctly identify the basis behind the complaint. After the problem has been identified, it is typical for the DLZ CML to begin a remediation effort and report the progress of this effort to the person or company making the complaint on the same day. A course of action is defined and a response acceptable to the client is established.

### ***Subcontracting***

In order to ensure the quality of external technical services used in accordance with providing services to a client, the DLZ CML requires that the subcontracted laboratory participates in the AMRL and CCRL inspection programs and/or proficiency sample program. The subcontracted laboratory must also be AMRL and/or CCRL certified. A list of all Approved Subcontractors can be found in Appendix IX.

When test results are received by the DLZ CML from the subcontracted laboratory, the results are first reviewed by the Manager or Laboratory Supervisor in order to ensure that the results are correct and that they contain the information requested by the client. If changes are necessary, the subcontractor is requested to make the changes. Once the test results are reviewed by the Manager or Laboratory Supervisor, then they forwarded to the client without modifications. A cover letter from the DLZ CML may accompany the test results in order to explain why the work was subcontracted to another laboratory.

**CONSTRUCTION MATERIALS LABORATORY  
QUALITY SYSTEM**

### ***Internal Quality System Review***

Several internal and external reviews of the DLZ Construction Materials Laboratory Internal Quality Control Program are conducted throughout the calendar year. In order to verify its Quality Control Program, the CML at DLZ participates in the U.S. Army Corps of Engineers Inspection Program wherein laboratory personnel, procedures, and equipment are periodically inspected by The U.S. Army Corps of Engineers. In addition, the CML at DLZ ascribes to the AMRL and CCRL proficiency programs for certain soils, aggregate, and concrete tests and also participates in the AMRL Inspection Program.

Internal and external reviews are conducted of the DLZ CML in order to ensure that established quality procedures are being followed. Specifically, these reviews cover:

1. Proficiency samples and reports.
2. On-site inspections and reports.
3. Quality System evaluations and reports.
4. Equipment calibration, verification, and inspection reports.
5. Technician training and records.
6. Technician evaluations and records.

Internal reviews are conducted once a year, during first quarter. External reviews are conducted during the AMRL on-site inspections.

All CML technicians are subject to continuous review and reinstruction by the CML Supervisor, field supervisory personnel, or Laboratory Geotechnician Level II or higher. When a deviation in procedure or results is observed, the technician responsible for the deviation is formally reinstructed and must again demonstrate competence in the relevant testing procedure.

The Manager and Laboratory Supervisor are responsible for ensuring that the Construction Materials Laboratory Quality System is reviewed on a regular basis. Copies of each Quality System Review are given to the Manager. The results of each Quality System Review are kept in a specific filing cabinet in the CML and are available for review by all DLZ employees as well as external individuals or organizations.

### ***Construction Materials Laboratory Accreditation's***

The Construction Materials Laboratory at DLZ is certified and accredited by the AASHTO Materials Reference Laboratory (AMRL). This certification and accreditation became effective January 1, 1998.

The AMRL Certificates are kept on file in the Department Manager's office. Copies of the Certificates are posted in the Construction Materials Laboratory.

## **APPENDIX I**

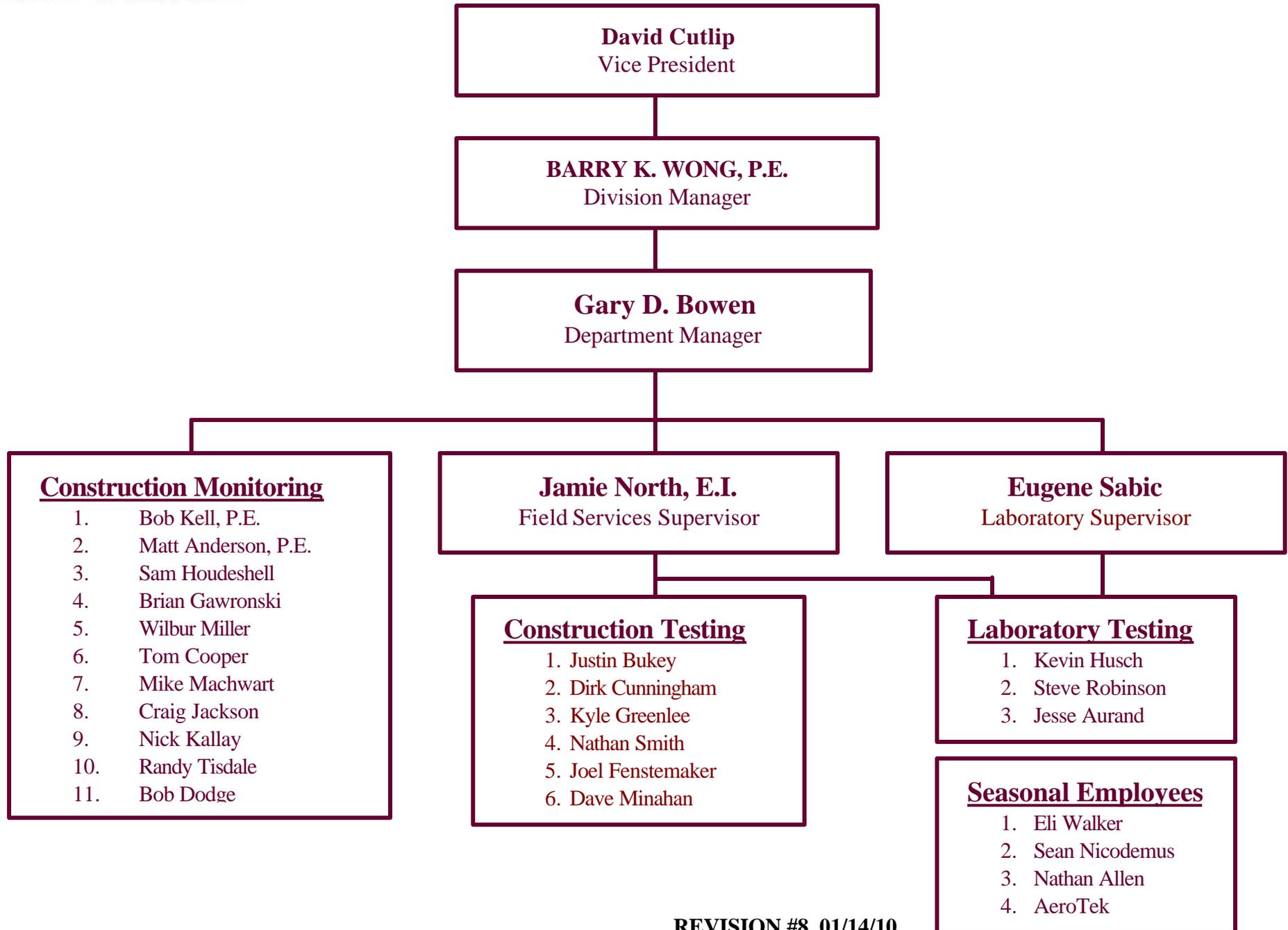
### **Division Organizational Chart**

**REVISION #8, 01/14/10**



# Construction Services

## Organizational Chart



## **APPENDIX II**

### **Construction Materials Laboratory Floor Plan**

**REVISION #8, 01/14/10**



## **APPENDIX III**

### **Position Descriptions**

**REVISION #8, 01/14/10**

**TITLE: Project Manager**

**REPORTS TO: Division Manager**

**RESPONSIBILITY:**

1. Responsible for managing, coordinating and administering the entire project from engineering and project planning procurement through construction and start-up.
2. Assisting business development in preliminary business development efforts.
3. Reviewing proposed contracts to determine significant parameters, time schedules and mileposts and defining the major task areas.
4. Monitors to assure schedules are met and work is performed within budget based on project planning.
5. As the principal contact with the client throughout the entire project, resolves problems and coordinates the final turnover of the project to the client.
6. Maintains management level relationships with members of other divisions and departments responsible for performing services in connection with the project.

**PRIMARY DUTIES:**

1. Directs the planning and development of the project scope, procedure, budget and overall project scope.
2. Reviews and approves all major change orders and periodically reviews change order controls to determine their adequacy and whether change orders are current and adequate.
3. Advises subordinates on professional development, recommends ways for them to advance.
4. Controls and/or reviews all project forecasts, schedules, cost estimates and reports. Is responsible for quality control over the complete project.
5. Coordinates all client contact and maintains working relationships at all levels throughout the period of the project including correspondence between the company and the client.
6. Ensures contractual commitments are met and the work is being performed according to the client's instructions.
7. Conducts periodic client meetings to review progress, discusses construction and engineering changes to project suggested by client, and resolves problems of coordination, schedule, and priority.
8. Prepares or directs the preparation of progress and special reports to staff and clients.
9. Obtains client's acceptance of work, coordinates completion of final project paperwork and preparation of final report.

**REVISION #8, 01/14/10**

10. Works with the accounting department to keep billings and receipts current on projects and clients under his/her responsibility.
11. Is a member of and works to be actively involved in civic and professional activities, societies and organizations.
12. Performs and has the authority to carry out the general functions inherent in a managerial position.
13. Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1. Doctoral Degree in profession, registered or licensed professional, 7 years related experience, or
2. Masters Degree in profession, registered or licensed professional, plus 8 years related experience, or
3. Bachelor's Degree, registered or licensed professional, plus 9 years related experience.

**TITLE:                    Engineer II**

**REPORTS TO:            Project Manager**

**RESPONSIBILITY:**

1.     Performs engineering and design assignments of a varied nature requiring a solid grasp of at least one engineering discipline and the exercise of professional judgment.
2.     Typically is given projects of moderate complexity and may have leadership responsibility for less experienced engineers or technicians. With close supervision, leadership levels can increase in complexity as training for more advanced management or technical responsibility.
3.     May be specializing in a single field, assuming lead roles in a narrow segment of a project and/or coordinating work of specialty groups related to his/her assigned segment of a project.
4.     Performs engineering assignments with general guidance and supervision.

**PRIMARY DUTIES:**

1.     Prepares reports and conducts economic and operating feasibility studies and other studies aimed at evaluating alternative systems, equipment, materials or engineering methods.
2.     Performs and directs others in the design of structures, systems and equipment items of moderate complexity.
3.     Directs engineers and technical support staff in the preparation of detailed drawings; reviews and checks finished drawings.
4.     Prepares contract specifications.
5.     Conducts analyses of engineering problems and design deficiencies; recommends action to engineering management.
6.     Prepares construction cost estimates.
7.     Prepares proposals establishing the scope of work, man-hour requirements, and schedules.
8.     Reviews contractor's shop drawing submittals.
9.     Assists in the preparation of client progress reports, as requested.
10.    Participates in technical development of personnel assigned to him/her.
11.    Performs construction monitoring.
12.    Coordinates field activities.
13.    Performs other duties as assigned.

**REVISION #8, 01/14/10**

**TITLE:**               **Engineer II (Cont.)**

**REPORTS TO:**       Project Manager

**RESPONSIBILITY:**

**JOB QUALIFICATIONS:**

1.     Bachelors Degree in engineer, plus 4 years experience, or
2.     Masters Degree, plus 3 years experience, or
3.     Doctoral Degree, plus 2 years experience.

Note: Professional Engineering registration is not required.

**REVISION #8, 01/14/10**

**TITLE:                    Engineer III**

**REPORTS TO:            Project Manager**

**RESPONSIBILITY:**

1.     Frequently assigned a lead role in a major segment of design projects where planning work is to be done; directing assigned personnel, meeting cost and time schedules and producing quality results are primary objectives.
2.     May be given difficult, complex, and demanding assignments calling for the application of specialized professional expertise which is performed independent of other personnel.
3.     May be responsible for managing, coordinating, and administering an entire project from engineering through construction and start-up.
4.     Operates with little guidance and direction in areas where precedents are not clearly established.
5.     Performs marketing and client development.

**PRIMARY DUTIES:**

1.     Prepares reports and conducts studies of new engineering methods, codes, processes, and materials. Conducts complex feasibility and operating studies and studies aimed at evaluating alternative systems, equipment, etc.
2.     Develops concepts and approaches to the solution of complex engineering problems.
3.     Directs engineers and technical support staff in the preparation of plans and specifications for major and complex structures, systems, and equipment.
4.     Prepares construction cost estimates.
5.     Performs construction monitoring.
6.     Coordinates field activities.
7.     Performs construction phase activities including bid evaluations and review of contractor's shop drawing submittals.
8.     Prepares proposals, establishing the scope of work, man-hour requirements, and schedules.
9.     Participates in technical development of personnel assigned to him/her.
10.    May be responsible for a project with the following duties:
  - Directs the planning and development of the project scope, procedure, budget and overall project scope.
  - Controls and/or reviews all project forecasts, schedules cost estimates and reports. Is responsible for quality control over the complete project.

**REVISION #8, 01/14/10**

- Coordinates all client contact and maintains working relationships at all levels throughout the period of the project.

**TITLE: Engineer III (Cont.)**

**REPORTS TO:** Project Manager

**RESPONSIBILITY:**

- Ensures contractual commitments are met and the work is being performed according to the client's instructions.
  - Conducts periodic client meetings to review progress, discusses construction and engineering changes to project suggested by client or recommended by our firm and resolves problems of coordination, schedule and priority.
  - Reviews and approves issuance of all design drawings, change orders and engineering specifications for construction, procurement or subcontract use as well as all bid analyses and recommendations for purchase.
  - Coordinates activities among the design groups and obtains the assistance of other corporate technical specialist groups.
  - Prepares or directs the preparation of progress and special reports to in-house management and clients.
  - Resolves field initiated questions pertaining to design or procurement. Visits field and participates in job site conferences.
  - Obtains client's acceptance of work, coordination completion of final project paperwork and preparation of final report.
  - Works with the accounting department to keep billings and receipts current on project and clients under his/her responsibility.
11. Performs other duties as assigned.

**JOB QUALIFICATIONS:**

Graduate of accredited College of Engineering with:

1. Bachelors Degree in engineering plus 8 years related experience, or
2. Masters Degree in engineering, plus 7 years related experience, or
3. Doctoral Degree in engineering, plus 6 years related experience.

Note: Professional Engineering registration required to enter position.

**REVISION #8, 01/14/10**

**TITLE:                   Geotechnical Laboratory Manager**

**REPORTS TO:           Project Manager / Division Manager**

**RESPONSIBILITY:**

1.     Coordinates all activities in the geotechnical laboratory. Hires and directs the training of laboratory geotechnicians. Implements and enforces safety procedures and company policies.
2.     Makes sure the laboratory runs smoothly and efficiently. Monitors activities to assure schedules are met.
3.     Assists business development efforts and marketing of geotechnical laboratory.

**PRIMARY DUTIES:**

1.     Supervises and schedules testing in the laboratory.
2.     Performs laboratory tests, when needed.
3.     Reviews all laboratory test results.
4.     Readily recognizes and corrects laboratory testing errors.
5.     Writes reports of test results and submits results to clients in a timely manner, or notifies persons requesting testing when work has been completed.
6.     Maintains all laboratory equipment.
7.     Reviews new test procedures and coordinates start up of new testing when required.
8.     Logs in all samples delivered to the laboratory for testing, and maintains a record of where all samples are temporarily stored.
9.     Participates in training development and performance evaluations of Laboratory Geotechnicians.
10.    Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     At least two years of post secondary education with 5 years of experience in laboratory soil testing, and
2.     Thorough knowledge of and is able to conduct all current laboratory tests (ASTM, AASHTO, Corps of Engineers, ODOT, etc.) and
3.     Certified NICET Level II in applicable subject areas. Proper State DOT certification, and
4.     One year experience as a Field Geotechnician I or equivalent, and
5.     Valid drivers license and willing to use personal vehicle for job-related work, as needed.

**REVISION #8, 01/14/10**

**TITLE:                   Laboratory Geotechnician III**

**REPORTS TO:           Geotechnical Laboratory Supervisor**

**RESPONSIBILITY:**

1.     Conducts and writes reports on soils, concrete, and asphalt laboratory testing.
2.     Schedules testing.

**PRIMARY DUTIES:**

1.     Directly conducts and supervises testing in a limited area or section of the laboratory.
2.     Writes reports of test results.
3.     Receives and maintains contacts with clients.
4.     Conducts some Level III\* laboratory testing.
5.     Assumes supervision of entire laboratory when lab supervisor is not present.
6.     Performs field quality control testing and inspection when required on limited basis.
7.     Initiates the training of technicians assigned to the same area or scope of the laboratory.
8.     Recognizes and corrects errors in test results.
9.     Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     At least two years of post secondary education, and
2.     Two years experience as a Laboratory Geotechnician I and two years as a Laboratory Geotechnician II, or equivalent, and
3.     Able to conduct all Basic, Level I and Level II tests, and
4.     Certified NICET Level II in either a construction materials testing or geotechnical subject, and
5.     One year experience as a Field Geotechnician I, or equivalent, and
6.     Valid driver's license and willing to use personal vehicle for job related work, as needed.

\* - See Construction Materials Laboratory - Testing Levels

**REVISION #8, 01/14/10**

**TITLE:                   Laboratory Geotechnician II**

**REPORTS TO:           Geotechnical Laboratory Supervisor**

**RESPONSIBILITY:**

1.     Conducts soils, concrete and asphalt laboratory testing.

**PRIMARY DUTIES:**

1.     Conducts all basic\* soils and concrete laboratory testing.
2.     Conducts all Level I\* laboratory testing.
3.     Conducts some of the Level II\* laboratory testing.
4.     Explains and demonstrates Basic and Level I laboratory skills to level I technicians, as assigned.
5.     Recognizes and reports errors in test results.
6.     Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     High school graduate or equivalent with two years experience as Laboratory Geotechnician I or equivalent, and,
2.     NICET Level I certification or equivalent, and
3.     Valid driver's license and willing to use personal vehicle for job related work, as needed.

\* - See Construction Material Laboratory - Testing Levels

**REVISION #8, 01/14/10**

**TITLE:                    Laboratory Geotechnician I**

**REPORTS TO:            Geotechnical Laboratory Supervisor**

**RESPONSIBILITY:**

1.        Soils and concrete laboratory testing.

**PRIMARY DUTIES:**

1.        Conducts all basic\* soils and concrete laboratory testing.
2.        Conducts some Level I\* laboratory testing.
3.        Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.        High school graduate or equivalent, and
2.        Valid driver's license and willing to use personal vehicle for job related work, as needed.

\* - See Construction Material Laboratory - Testing Levels

**REVISION #8, 01/14/10**

**TITLE:                   Field Services Manager**

**REPORTS TO:           Division Manager**

**RESPONSIBILITY:**

1.     Responsible for managing, coordinating and administering the entire project from engineering and project planning procurement through construction and start-up.
2.     Assisting business development in preliminary business development efforts.
3.     Reviewing proposed contracts to determine significant parameters, time schedules and mileposts and defining the major task areas.
4.     Monitors to assure schedules are met and work is performed within budget based on project planning.
5.     As the principal contact with the client throughout the entire project, resolves problems and coordinates the final turnover of the project to the client.
6.     Maintains management level relationships with members of other divisions and departments responsible for performing services in connection with the project.

**PRIMARY DUTIES:**

1.     Directs the planning and development of the project scope, procedure, budget and overall project scope.
2.     Reviews and approves all major change orders and periodically reviews change order controls to determine their adequacy and whether change orders are current and adequate.
3.     Advises subordinates on professional development, recommends ways for them to advance.
4.     Controls and/or reviews all project forecasts, schedules, cost estimates and reports. Is responsible for quality control over the complete project.
5.     Coordinates all client contact and maintains working relationships at all levels throughout the period of the project including correspondence between the company and the client.
6.     Ensures contractual commitments are met and the work is being performed according to the client's instructions.
7.     Conducts periodic client meetings to review progress, discusses construction and engineering changes to project suggested by client or recommended by SEG and resolves problems of coordination, schedule and priority.
8.     Prepares or directs the preparation of progress and special reports to staff and clients.
9.     Obtains client's acceptance of work, coordinates completion of final project paperwork and preparation of final report.

**REVISION #8, 01/14/10**

**TITLE:                   Field Services Manager (Cont.)**

**REPORTS TO:           Division Manager**

**PRIMARY DUTIES:**

10.   Works with the accounting department to keep billings and receipts current on projects and clients under his/her responsibility.
11.   Is a member of and works to be actively involved in civic and professional activities, societies and organizations.
12.   Performs and has the authority to carry out the general functions inherent in a managerial position.
13.   Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.    Doctoral Degree in profession, registered or licensed professional, 7 years related experience, or
2.    Masters Degree in profession, registered or licensed professional, plus 8 years related experience, or
3.    Bachelor's Degree, registered or licensed professional, plus 9 years related experience.

**REVISION #8, 01/14/10**

**TITLE: Geotechnical Field Supervisor**

**REPORTS TO: Project Manager**

**RESPONSIBILITY:**

1. Coordinates all activities in the field. Hires and directs the training of field geotechnicians. Implements and enforces safety procedures and company policies.
2. Makes sure field operations run smoothly and efficiently. Monitors activities to assure schedules are met.
3. Assists business development efforts and marketing of geotechnical field services.

**PRIMARY DUTIES:**

1. Supervises and schedules field activities.
2. Performs field tests when needed.
3. Reviews all field test results.
4. Readily recognizes and corrects field errors.
5. Writes reports of test results and submits results to clients in a timely manner, or notifies person requesting testing when work has been completed.
6. Maintains all field equipment.
7. Reviews new procedures and coordinates start up of new activities when required.
8. Participates in training, development and performance evaluations of field geotechnicians.
9. Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1. Bachelors degree in Civil Engineering, and
2. Three years experience in field quality control testing, and
3. One year experience in laboratory testing, and
4. Valid drivers license and willing to use personal vehicle for job-related work, as needed.

**REVISION #8, 01/14/10**

**TITLE:                   Field Geotechnician III**

**REPORTS TO:           Geotechnical Field Supervisor**

**RESPONSIBILITY:**

1.     Field quality control testing on all types of projects.
2.     In charge of inspection and testing for one or more projects.
3.     Direct interaction with client on most projects.

**PRIMARY DUTIES:**

1.     Performs all field testing (concrete, compaction and related tests) efficiently and in complete accordance with specified procedures and standards.
2.     Performs structural steel, reinforcing steel, footing excavation and drilled pier installation monitoring as required.
3.     Performs other related types of inspection with minimal instruction.
4.     Performs all basic\* laboratory tests (grain size analysis, plasticity, and Proctors, including preparation and moisture contents) with no supervision.
5.     Initiates interaction with clients and contractors as well as reading plans and specifications to determine required testing and monitoring services.
6.     Recognizes unusual site circumstances or faulty test results quickly and makes appropriate corrections.
7.     Provides constructive suggestions to solve most common problems encountered in the field, that may have caused failing test.
8.     Trains new technicians in all basic laboratory and field tests as required.
9.     Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     High School graduate or equivalent with five years experience (supervisor experience preferred) as Geotechnician I and II or equivalent and capable of performing all routine laboratory and field testing, and
2.     ACI and NICET level II Certification, and
3.     Proper state Department of Transportation certifications (asphalt and P.C. concrete, field density testing, etc.)

\* - See Construction Material Laboratory - Testing Levels

**REVISION #8, 01/14/10**

**TITLE:                   Field Geotechnician II**

**REPORTS TO:           Geotechnical Field Supervisor**

**RESPONSIBILITY:**

1.     Field quality control testing on various projects.
2.     Generally runs tests and reports results to supervisor or designated on-site client representative.

**PRIMARY DUTIES:**

1.     Performs P.C. concrete and compaction testing in accordance with specified procedure and standards. Performs all related field and laboratory tests.
2.     Performs asphalt testing and other types of inspection as requested.
3.     Maintains all records of test results and daily reports.
4.     Immediately reports unsatisfactory test results on-site conditions to on-site client representative and as soon as practical to the field services supervisor.
5.     Recognizes unusual test results or site conditions, reports these in a timely manner. Offers constructive suggestions for possible corrections.
6.     Assists in training new technicians in basic field testing as requested.
7.     Determines specified testing and inspection requirements from plans and specifications as needed.
8.     Conducts all basic\* soils and concrete laboratory testing.
9.     Conducts all Level I\* laboratory testing.
10.    Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     High school graduate, or equivalent with two years experience as Geotechnician I, or equivalent and
2.     NICET Level I certification or equivalent, and
3.     Valid driver's license and willing to use personal vehicle for job related work, as needed.

\* - See Construction Material Laboratory - Testing Levels

**REVISION #8, 01/14/10**

**TITLE:                   Field Geotechnician I**

**REPORTS TO:           Geotechnical Field Supervisor**

**RESPONSIBILITY:**

1.     Field quality control testing on various projects.
2.     Generally runs tests and reports results to supervisor or designated on-site client representative.

**PRIMARY DUTIES:**

1.     Performs P.C. concrete and/or compaction testing and other related field tests in accordance with specified procedure and standards.
2.     Maintains records of tests and daily reports.
3.     Immediately reports unsatisfactory test results to on-site client representative and as soon as practical to the field services supervisor.
4.     Recognizes unusual test results or site conditions and reports these in a timely manner to supervisor and others as necessary.
5.     Conducts all basic\* soils and concrete laboratory testing.
6.     Performs other duties as assigned.

**JOB QUALIFICATIONS:**

1.     High school graduate, or equivalent, and
2.     Valid driver's license and willing to use personal vehicle for job related work, as needed.

\* - See Construction Material Laboratory - Testing Levels

**REVISION #8, 01/14/10**

*Construction Materials Laboratory Testing Levels*

**BASIC LEVEL**

Standard Proctor  
Plasticity Index (PI)  
Water Content (oven)  
Sieve Gradation  
Sample Preparation  
Safety Procedures  
Concrete Cylinders: strip, cap, break

**LABORATORY LEVEL I**

Modified Proctor  
One-Point Proctor Determination  
Water Content (microwave oven)  
Computer Work (enter lab test data)  
Grain Size Analysis  
Hydrometer  
Specific Gravity of Soil  
Unit Weights of Cubes and Cylinders  
Proper Sample Storage

**LABORATORY LEVEL II**

Prepare Proctor Samples  
Relative Density  
Unconfined Compression Strength  
Remolding Soil Samples  
Loss on Ignition (LOI)  
Bulk Specific Gravity and Absorption of Aggregates  
Solubility Test  
Calibrate Equipment  
Extrude and Trim Press Tube Samples  
Consolidation  
Flexural Strength of Beams  
Asphaltic Concrete Extraction and Gradation  
Chloride Ion Test

**LABORATORY LEVEL III**

Rock Core: prepare specimens,  
compressive strength,  
rebar pullout test  
Shrinkage Limit  
Pick Tests on Aggregate Quality  
Sodium Sulfate Soundness  
California Bearing Ratio (CBR)  
Soil Visual Classification  
Concrete Mix Design  
Asphalt Mix Design  
Permeability Test: flexible wall,  
constant head  
Triaxial Test  
Direct Shear  
Marshal Stability and Flow  
Slake Durability Index  
Total Hardness  
Bulk Specific Gravity of Asphalt  
Theoretical Maximum Specific  
Gravity of Asphalt (Rice Test)

**REVISION #8, 01/14/10**

Carbonation Testing (Phenolphthalein Testing)

**REVISION #8, 01/14/10**

**APPENDIX IV**

Resumes for Barry Wong  
Jamie North  
Eugene Sabic

**REVISION #8, 01/14/10**

**INSERT RESUMES FOR B. WONG, J. NORTH, AND E. SABIC**

**REVISION #8, 01/14/10**

**APPENDIX V**

**Master Task Sign-Off Sheet**

**REVISION #8, 01/14/10**

Tech Name	TASK	ASTM	AASHTO
<b>CONCRETE</b>			
<b>Lab</b>			
	CONCRETE - Breaking	C39	T22
	- Capping	C617	T231
	- Stripping & Marking		
	CONCRETE BEAMS - Breaking	C78	T97
	- Unit Weight		
	MORTAR CUBES - Breaking		
	- Unit Weight		
	Chloride Ion		
	Diamond Sawing		
<b>Field</b>			
	Making and Curing Concrete Test Specimen	C31	T23
	Unit Wt., Yield & Air Content (Gravimetric)	C138	T121
	Slump	C143	T119
	Air Content (Volumetric)	C173	T196
	Air Content (Pressure Method)	C231	T152
<b>CURED - IN - PLACE PIPE (CIPP)</b>			
	Preparing / Cutting		
	Measuring		
	Breaking the Specimen		
	Reporting		
<b>ASPHALT</b>			
	Quantitative Extraction	D2172	T164
	Mechanical Analysis of Extracted Aggregate	D5444	T30
	Asphalt Content of HMA by the Ignition Method (NCAT)	D6307	T308
	Asphalt Content of Bituminous Mixtures by the Nuclear Method	D4125	T287
	Maximum Specific Gravity (Rice)	D2041	T209
	Bulk Specific Gravity of Bituminous Mixture	D2726	T166
<b>SOILS</b>			
	California Bearing Ratio	D1883	T193
	Consolidation	D2435	T216
	Direct Shear	D3080	T236
	Setting Up Perm/Triaxial Sample		
	Remolding Soil Samples		
	Press Tube Extraction		
	TRIAXIAL TEST - UU	D2850	T296
	- CIU	D4767	T297
	Unconfined Compression Test	D2166	T208
	GRADATION - Dry (small)		
	- Dry (Large)		
	- "Rotap"		
	- Wash Sieve	C136	T27
	- Hydrometer		
	Atterberg Limit	D4318	T89
	Loss on Ignition		
	Obtain Soil Samples for Testing		
	PERMEABILITY TEST - Soil	D5084	
	- Granular		
	pH of Soils		
	Splitting and Quartering		
	PROCTOR CURVE - Setting Up	D698/D1557	T99 / 180
	- Pounding		
	- One Point		
	Relative Density		
	Sodium Sulfate Soundness		
	Shrinkage Limit		
	SPECIFIC GRAVITY - Soil	D854	T100
	- Aggregate	C127 / C128	T84 / 85
	Water Content	D2216	T265

**REVISION #8, 01/14/10**

| Weighing Oven Contents | | |

## **APPENDIX VI**

### Equipment

**REVISION #8, 01/14/10**

## **APPENDIX VII**

### **In-House Calibration Procedures:**

- Procedure #1 Ovens**
- Procedure #2 Vacuum System**
- Procedure #3 Manual Hammers**
- Procedure #4 Conical Mold, Tamper**
- Procedure #5 Straightedges**
- Procedure #6 Grooving Tool**
- Procedure #7 Liquid Limit Device**
- Procedure #8 Hydrometer Bulbs**
- Procedure #9 California Bearing Ratio Equipment**
- Procedure #10 Sodium Sulfate Containers**
- Procedure #11 Compaction Molds**
- Procedure #12 Mechanical Sieve Shakers**
- Procedure #13 One-Dimensional Consolidation Load Indications**
- Procedure #14 Unit Weight Measure**
- Procedure #15 Wire Cloth Sieves**
- Procedure #16 Laboratory Moisture Cans**
- Procedure #17 Digital Calipers**
- Procedure #18 Mechanical vs. Manual Marshall Hammers**
- Procedure #19 NTO Burn-off Oven & Aggregate Correction Factor**
- Procedure #20 In-house Verification of Dial Indicators**
- Procedure #21 Marshall Molds**
- Procedure #22 Verify Thermometer Accuracy**

## **In-House Calibration and Verification Procedure : #1**

Equipment Checked: Ovens

Purpose: This procedure provides instructions for verifying and adjusting the temperature of general purpose drying ovens.

Equipment Required:

- 1) A calibrated datalogger graduated in 0.1°C increments having a range which includes the temperature range to be verified.
- 2) A calibrated datalogger graduated in 0.1°C increments having a range which includes the temperature range to be verified.

Tolerance: Ovens shall be capable of maintaining a constant temperature range specified in the appropriate test method.

### Procedure:

1. The first thermometer is to be permanently located in the vent hole on top of the oven. This permanent thermometer shall be positioned so that the thermometer bulb is located several inches inside of the oven while the temperature scale on the thermometer is located outside of the oven. The thermometer shall be positioned in such a manner as to allow reading of the temperature without moving the thermometer or opening the oven doors.
2. Each morning prior to opening the oven doors, read and record the oven temperature to the nearest 1 C.
3. Adjust the oven temperature as needed if the observed temperature reading is outside of the specified tolerance.
4. For any drying ovens which have built-in temperature monitoring equipment, the temperature determined by the built-in monitoring equipment shall be compared to the temperature determined by the permanent thermometer. Any discrepancies shall be reported to the laboratory supervisor. The temperature determined by the permanent thermometer shall be taken as the true oven temperature.
5. Every four months the calibrated datalogger probe shall be placed inside the oven and set to record temperatures over a four hour period. A graph shall be constructed on a time/temperature basis. Several readings from the calibrated datalogger, permanent

**REVISION #8, 01/14/10**

thermometer, and built-in temperature monitoring equipment shall be recorded throughout the day. The temperature determined by the calibrated datalogger shall be taken as the true oven temperature. The calibrated datalogger shall then be used to verify the readings of the permanent thermometer as well as the built-in temperature monitoring equipment. Calibration curves may be constructed for the Permanent thermometer and the built-in temperature monitoring equipment if desired.

## **In-House Calibration and Verification Procedure : #2**

Equipment Checked: Vacuum System (AASHTO T100, ASTM D854)

Purpose: This procedure provides instructions for checking the vacuum pressure of the vacuum system.

Equipment Required:

- 1) Absolute pressure gauge capable of measuring the vacuum pressure to the nearest 0.25 psi (2 kPa).
- 2) Mercury manometer capable of measuring the vacuum pressure to the nearest 0.1 cm.

Tolerance: Vacuum system shall be capable of applying the vacuum pressure specified in the appropriate test method.

### Procedure:

1. The absolute pressure gauge is to be permanently attached to the vacuum system. This will allow reading and adjusting of the vacuum pressure during each use.
2. Connect the vacuum system as necessary to the appropriate test equipment.
3. Turn on the vacuum system.
4. Adjust the vacuum pressure as necessary by opening or closing the supply lines and the pet-cock on the vacuum system in order to obtain the desired vacuum pressure.
5. Every 6 months, connect the mercury manometer to the vacuum system.
6. Measure and record the vacuum pressure as determined by the mercury manometer by opening or closing the supply lines and the pet-cock. Record this vacuum pressure to the nearest 0.1 cm of Mercury.
7. The vacuum pressure determined by the mercury manometer shall be taken as the true vacuum pressure. Adjust the absolute pressure gauge as necessary so that it corresponds with the vacuum measured by the mercury manometer. A calibration curve may be constructed for the absolute pressure gauge if desired.

**REVISION #8, 01/14/10**

### **In-House Calibration and Verification Procedure : #3**

Equipment Checked: Manual Hammers (AASHTO T99, T180, ASTM D698, D1557)

Purpose: This method provides instructions for checking the critical dimensions of the proctor hammers.

Equipment Required:

1. Calipers readable to 0.01 mm.
2. Steel or aluminum ruler readable to 1 mm.
3. Balance, capacity 5 kg, readable to 1 g.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

Procedure:

1. Measure and record the diameter of the rammer face determined by taking six readings 120° apart using the calipers. Record the measurements to the nearest 0.01 mm.
2. Pull up the handle and measure and record the drop height of the hammer to the nearest 1 mm. Determine this height inside the guide-sleeve using the steel or aluminum ruler.
3. Remove the hammer from the guide-sleeve. Determine and record its mass to the nearest 1 g.
4. Measure and record the diameters of the vent holes near the end of the hammer. Record the measurements to the nearest 0.01 mm.

## **In-House Calibration and Verification Procedure : #4**

Equipment Checked: Conical Mold, Tamper (AASHTO T84, ASTM C128)

Purpose: This method provides instructions for checking the critical dimensions of the sand cone and tamper used in the above test method.

### Equipment Required:

1. Calipers readable to 0.01 mm.
2. Balance, 500 g capacity, readable to 0.1 g.

Tolerance: Equipment shall meet the dimensional tolerances specified in the test method.

### Procedure: **(Cone)**

1. Measure the inside diameter at the top of the cone to the nearest 0.01 mm by taking six readings 120° apart using the calipers. Record the results.
2. Invert the cone and repeat step 1.
3. Place the cone on a flat glass surface. Measure and record the depth of the cone to the nearest 0.01 mm by taking three readings 120° apart using the calipers. Record the results.
4. Measure the thickness of the cone to the nearest 0.01 mm by taking three readings 120° apart at the top of the cone and three readings 120° apart at the bottom of the cone using the calipers. Record the results.

### **(Tamper)**

1. Measure and record the diameter of the tamping face to the nearest 0.01 mm by taking three readings 120° apart using the calipers. Record the results.
2. Determine and record the mass of the tamper to the nearest 0.1 g.

## **In-House Calibration and Verification Procedure : #5**

Equipment Checked: Straightedges (AASHTO T99, T180, ASTM D698, D1557)

Purpose: This method provides instructions for checking the critical dimensions of the straightedges.

Equipment Required:

1. Calipers readable to 0.01 mm.
2. Steel or aluminum ruler readable to 1 mm.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

Procedure:

1. Measure and record the thickness of the straightedge at no less than four locations along its length to the nearest 0.01 mm using the calipers.
2. Measure and record the length of the straightedge at no less than two locations along its length to the nearest 1 mm using the ruler.
3. Check straightedge for planeness using glass plate must be  $\pm 0.005$  inches over total length.

**REVISION #8, 01/14/10**

**In-House Calibration and Verification Procedure : #6**

Equipment Checked: Grooving Tool (AASHTO T89, ASTM D4318)

Purpose: This method provides instructions for checking the critical dimensions of the grooving tool.

Equipment Required: Calipers readable 0.01 mm.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

Procedure (AASHTO T89 Grooving Tool):

1. Measure and record the critical dimensions "a", "b", "c", and "d" (as shown in AASHTO T89 - Figure 1) to the nearest 0.01 mm using the calipers.

Procedure (ASTM D4318 Grooving Tool):

1. Measure and record the critical dimensions ? A? , ? B? , ? C? , ? D? , and ? K? (as shown in ASTM D4318 - Figure 2) to the nearest 0.01 mm using the calipers.

**In-House Calibration and Verification Procedure: #7**

Equipment Checked: Liquid Limit Device (AASHTO T89, ASTM D4318)

Purpose: This method provides instructions for checking the critical dimensions of the liquid limit device.

Equipment Required: Calipers readable to 0.01 mm.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

Procedure:

1. Measure and record the critical dimensions of the liquid limit device (specified in the applicable test method) to the nearest 0.01 mm using the calipers.
2. Adjust the height of the drop of the cup as necessary to meet the critical dimensions specified in the applicable test method.

## **In-House Calibration and Verification Procedure : #8**

Equipment Checked: Hydrometer Bulbs (AASHTO T88, ASTM D422, E100)

Purpose: This method provides instructions for checking the critical dimensions of the hydrometer bulbs.

Equipment Required: Calipers readable to 0.01 mm, constant temperature bath, and thermometer.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

### Procedure:

1. Measure and record the critical hydrometer dimensions (specified in the applicable test method) to the nearest 0.01 mm using the calipers.
2. Record measurements onto form and compare with critical dimensions supplied in AASHTO T-88.
3. Fill 1 liter graduate with 125 mL sodium Hex-Ametaphosphate and dilute to 1 liter mark.
4. Shake solution to fully disperse sod-hex in 1 L graduate.
5. Place graduate into constant temperature bath with heater, ice may be needed to for lower temperature range.
6. Take readings of sod-hex, water solution at various temperatures from 15°C to 30°C with hydrometer bulb.
7. Determine hydrometer bulb composite correction value at 20°C (Normally will be -2.8 to -3.5).
8. Apply results to grain-size reduction program.
9. Produce chart for file displaying the Temperature vs. Readings.

**REVISION #8, 01/14/10**

## **In-House Calibration and Verification Procedure : #9**

Equipment Checked: CBR Molds, Surcharge Weights & Spacer Disks.

Purpose: This method is used to calibrate the CBR Molds, Surcharge Weights & Spacer Disks.

Equipment Required: Scale with capacity of 30lbs and readable to 0.01lbs, Digital calipers readable to 0.01in, grease, CBR spacer, and Thermometer readable to 1°C.

Tolerance: CBR Molds, Surcharge Weights & Spacer Disks should be checked every year or if there is reason to question the accuracy.

### Procedure:

1. Assemble the CBR mold with the spacer inside of the mold, but without the collar of the mold attached.
2. Measure and record the height of the CBR mold at six evenly spaced intervals around the top of the mold, down to the top of the spacer.
3. Measure and record the interior diameter of the CBR mold at six evenly spaced intervals around the mold.
4. Rotate vertically the CBR mold (so the top is now the bottom), leaving the spacer alone and repeat steps #2 & 3.
5. Apply a “thick” coating of grease between the CBR spacer and the inside of the bottom of the CBR mold.
6. Measure and record the weight of the empty greased CBR mold and spacer with a glass plate on top of it.
7. Fill the greased CBR mold and spacer with water at a temperature of 20 to 25 °C. Using the glass plate as a screed, make sure that the CBR mold is full of water and that there are no leaks.
8. Measure and record the weight of the greased CBR mold and spacer with a glass plate on top of it and full of water. Confirm that the water temperature remained within 20 to 25 °C.
9. Using the density of water at the recorded temperature, determine and record the volume of the CBR mold.

10. Using the linear measurements, compare the CBR mold(s) with the ASTM & AASHTO specifications. The volume will be determined by the water filling method.
11. Inspect the holes on the bottom of the CBR mold to ensure they are not clogged, and clear any holes that appear to be clogged.
12. Measure and record the diameter of the slotted & anular CBR surcharge weight(s).
13. Measure and record the weight of the slotted & anular CBR surcharge weight(s).
14. Measure and record the diameter and thickness of the CBR spacer disk(s).
15. Measure and record the weight of the of the CBR spacer disk(s).
16. Compare measurements of the CBR surcharge weight(s) and the CBR spacer disk(s) with the ASTM and AASHTO specifications.

## **In-House Calibration and Verification Procedure: #10**

Equipment Checked: Sodium Sulfate Containers (AASHTO T104, ASTM C88)

Purpose: This method provides instructions for checking the physical condition and critical dimensions of the sodium sulfate containers used in the above test methods.

Equipment Required:

1. Calipers readable to 0.01 mm (for No. 8 sieve).
2. Eye comparator with a 0.1 mm scale (for No. 60 sieve).

Tolerance: Equipment shall meet the dimensional tolerances specified in the appropriate test method.

Procedure:

1. The DLZ CML uses a No. 8 sieve and a No. 60 sieve for the sodium sulfate containers.
2. Inspect the general condition of the sieves. Check the frame and solder joints for cracks or holes.
3. Make sure the sieves have appropriate labels.
4. Check for tightness of the wires on each sieve.
5. Verify the sieve size openings as dictated in ASTM Procedure E11.

## **In-House Calibration and Verification Procedures: #11**

Equipment Checked: Compaction Molds (T99/D698, T180, D1557)

Purpose: This method provides instructions for checking the critical dimensions of the compaction molds.

### Equipment Required:

1. Calipers having a measuring range of at least 0 to 6 inches and readable to at least 0.001 in.
2. Glass plates about 8 in<sup>2</sup> by ¼ inch thick.
3. Balance, capacity 5 kg, readable to 1 g.
4. Thermometer with measuring range of 0 °C to 50 °C and 0.5 °C graduations.

Tolerance: Equipment shall meet the dimensional tolerances specified in the applicable test method.

### Procedure:

1. Place the mold and bottom glass plate on a level surface and fill with water slightly above the top of the mold.
2. Slide top plate over the top of mold ensuring that mold is completely full.
3. Dry any excess water and record the mass of the mold, plates, and water.
4. Determine the temperature of the water and use the density to calculate the volume of the mold.
5. Using the calipers obtain six diameter measurement from the top and bottom of the mold equally spaced from each other.
6. Using the calipers obtain three height measurements from equally spaced locations.
7. Calculate the volume of the mold and compare to the volume obtained based on steps 1-4.

## **In-House Calibration and Verification Procedure: #12**

Equipment Checked: Mechanical Sieve Shakers

Purpose: This method is used to verify the Mechanical Sieve Shakers.

Equipment Required: Scale with capacity of 5000g and readable to 0.1g, Oven capable of maintaining  $110 \pm 5^{\circ}\text{C}$ , Timer, Wire brushes, Paint Brushes, Set of verified 8" sieves and Set of verified 12" sieves.

Tolerance: Mechanical Sieve Shakers should be checked every year or if there is reason to question the accuracy.

### Procedure:

1. Select sample for sieving. If testing 8" sieves select a sample of approximately 300-500g. If testing 12" sieves select a sample approximately 1000-1500g.
2. Oven dry the test specimen at  $110 \pm 5^{\circ}\text{C}$  to a constant mass.
3. Hand sieve the test specimen to refusal, meaning that upon hand sieving by the prescribed method outlined in ASTM C-136 less than 1% passes any of the test sieves after one minute of additional hand sieving.
4. Re-combine the hand sieved test specimen & place it into the sieve set for the mechanical sieve shaker to be tested.
5. Estimate a sieving time and set the sieve shaker to the estimated time. Run sieve shaker until it stops.
6. Compare the mechanical sieved masses retained/passing with the original hand-sieved results.
7. If the mechanically sieved and subsequently hand-sieved percent retained is less than the original hand-sieved results, then either re-hand-sieve the material and start over or discard the sample and start over. This would mean that the original time estimated for mechanical sieve shaker was too long and some aggregate breakdown may have occurred.

8. Continue to increase sieving time until less than 1% of each sieve matches the original hand sieved results.
9. If the mechanically sieved and subsequently hand-sieved percent retained is more than the original hand-sieved results, then re-combine the sample.
10. Re-sieve the sample and increase the sieving time by one minute.
11. Repeat steps #8 –10 until the hand-sieved and mechanically sieved results match, within 1%.
12. Repeat steps #1-11 for each type of aggregate necessary.

### **In-House Calibration and Verification Procedure: #13**

Equipment Checked: One-Dimensional Consolidation Load Indications (T216/D2435)

Purpose: This method provides instructions for verifying the load indications for the consolidometer.

Equipment Required:

1. Calipers readable to .001 inches.
2. Yardstick
3. Balance

Tolerance: The load device should be capable of maintaining the specified loads for long periods of time with an accuracy of + or – 0.5% of the applied load.

Procedure:

1. Measure the distance of the entire arm “ $x_1$ ”.
2. Measure the distance of the arm from the fulcrum to where the normal force is exerted “ $x_2$ ”.
3. Obtain the actual weight of the weights used to apply the load.
4.  $N = W (x_1/x_2)$
4. Calculate the normal force and use it with the sample diameter to calculate the actual load being applied.

## **In-House Calibration and Verification Procedure: #14**

Equipment Checked: Unit Weight Measure (T19/C29)

Purpose: This method provides instructions for checking the volume of unit weight measure.

Equipment Required:

1. Scale, Accurate and readable to 0.01 lb.
2. Temperature Recording Device – Accurate to 0.1°C, syringe or squirt bottle
3. Appropriate sized glass plate, water at a temperature between 20-30°C

Tolerance: Measures should be calibrated annually or whenever there is a reason to question the accuracy.

Procedure:

1. Weigh the unit weight measure with the appropriate size glass plate and record on the worksheet.
2. Fill the unit weight measure level full with water and use the glass plate as a screed, work out any air bubble.
3. Wipe off any excess water from the scale, bottom of the measure, and from around the glass plate.
4. Weigh the weight of the measure, water, and glass plate and record on the worksheet.
5. Record the temperature of the water inside of the measure using the temperature recording device, and record on the worksheet.
6. Subtract the empty weight of measure and glass plate from the full weight of the measure and glass plate, and record on the worksheet.
7. Using an interpolated value from the chart, or a table generated from the chart provided in ASTM C-29/2M determine the density of the water at the recorded temperature.
8. Divide the mass of the water to fill the measure by the density of water at the recorded temperature and record. This is the volume of the measure.

9. Paint or mark with permanent marker the volume of the measure on the unit weight measure.

**In-House Calibration and Verification Procedure: #14 cont.**

Equipment Checked: Unit Weight Measure (T19/C29)

NOTE: Optionally it may be desired to put a multiplying factor on the measure, this would be equal to:  
1/volume of the measure.

## **In-House Calibration and Verification Procedure: #15**

Equipment Checked: Wire Cloth Sieves (ASTM E 11-04 / AASHTO M 92-05)

Purpose: This method provides instructions for checking the opening on the sieves.

Equipment Required: Digital Calipers, readable to 0.001 inches.

Tolerance: Sieves should be checked every 6 months or if there is reason to question the accuracy.

Procedure (Sieves down to #5):

1. Measure all openings of the sieves, with 30 or less openings and record.
2. Measure 30 openings of the sieves that have 30 or more openings.

Procedure (#4 to #270 sieves):

1. Visually inspect all numbered sieves.
2. Make record of visual inspection.

## **In-House Calibration and Verification Procedure: #16**

Equipment Checked: Laboratory Moisture Cans

Purpose: This method provides data used to provide tare weights for cans that are used to create a list for laboratory use and various spreadsheets.

Equipment Required:

1. Laboratory scale, read and accurate to 0.01 grams, with a capacity of 100 grams.
2. Paint
3. Cleaning equipment

Tolerance: All cans should be checked every year or if there is reason to question the accuracy.

Procedure:

1. Thoroughly clean and remove asphalt tar, soil or any other product.
2. Re-paint the cans as needed.
3. Replace missing or damaged cans.
4. Re-weigh ALL cans.
5. Create can list and post.
6. Update all spreadsheets using new can weights.

**In-House Calibration and Verification Procedure: #17**

Equipment Checked: Digital Calipers

Purpose: This method is used to verify the accuracy of the digital calipers.

Equipment Required: NIST traceable gauge blocks.

Tolerance: Digital calipers should be checked every 6 months or if there is reason to question the accuracy.

Procedure:

1. Measure eight (8) NIST gauge blocks with the calipers and record the results.
2. Compare results.
3. If necessary, correction factor is developed for each individual caliper tested.

## **In-House Calibration and Verification Procedure: #18**

Equipment Checked: Mechanical vs. Manual Marshall Hammers

Purpose: This method is used to verify the accuracy of the mechanical marshall hammers (for 4" molds only).

Equipment Required: Oven capable of maintaining  $125 \pm 5^{\circ}\text{C}$  ( $257 \pm 9^{\circ}\text{F}$ ), Scale with capacity of 30lbs and readable to 0.01lbs, Calipers readable to 0.01in., Ruler readable to 1/64in, Thermometer readable to  $1^{\circ}\text{C}$ .

Tolerance: Hammers should be checked every year or if there is reason to question the accuracy.

### Procedure:

1. Set the oven temperature to the indicated mix temperature, if the mix temperature is not available then use  $257 \pm 9^{\circ}\text{F}$  as a default temperature.
2. Place samples into metal containers and heat to mix temperature, or default temperature.
3. Load approximately 1150 to 1200 grams into a 4" Marshall Mold, depending on the type of mix. Be sure the sample and equipment is  $257 \pm 9^{\circ}\text{F}$  at the start of the test.
4. One at a time, compact three Marshall Pills with the Manual Marshall Apparatus, one at 35 blows, one at 50 blows and one at 75 blows.
5. One at a time, compact three Marshall Pills with the Mechanical Marshall Apparatus, one at 35 blows, one at 50 blows and one at 75 blows.
6. Record drop heights and hammer weights for both apparatus's.
7. Record linear measurements along with weights of all six pills.
8. Calculate the unit weight of all six pills.
9. Perform BSG on all six pills.
10. Compare the results of the three pills performed with the Manual Marshall Apparatus with the three pills performed with the Mechanical Marshall Apparatus.
11. Repeat above steps for each mix.

12. The results from step #5 for the three pills performed with the Mechanical Marshall Apparatus must be within 2% of the three pills performed with the Manual Marshall Apparatus.
13. The average of the BSG's from the three pills performed with the Mechanical Marshall Apparatus must be within 2% of the average of the BSG's from the three pills performed with the Manual Marshall Apparatus.

## **In-House Calibration and Verification Procedure: #19**

Equipment Checked: NTO Burn-off Oven & Aggregate Correction Factor

Purpose: This method is used to determine the NTO and Aggregate Correction Factors

Equipment Required: NTO burn-off oven with internal scale, Set of verified sieves, Mixing tools, Mixing bowl, Burn-off baskets, Oven capable of maintaining  $125 \pm 5^{\circ}\text{C}$  ( $257 \pm 9^{\circ}\text{F}$ ), Scale with capacity of 5000g and readable to 0.1g, Thermometer readable to  $1^{\circ}\text{C}$ .

Tolerance: NTO Burn-off Oven & Aggregate Correction Factors should be checked for each individual mix design, every year or if there is reason to question the accuracy.

### Procedure:

1. Turn on NTO Burn-off Oven and set default burn profile to 75 minutes.
2. Set the oven temperature to the indicated mix temperature, if the mix temperature is not available then use  $257 \pm 9^{\circ}\text{F}$  as a default temperature.
3. Make five graded samples of aggregate to specific batch proportions. Three samples are to be mixed to the desired AC percentage, one sample will be kept “blank” (no AC added to aggregate) and one sample will be kept for a “butter” mix.
4. Load the “blank” sample into the NTO Burn-off Oven at the default burn profile and record results when completed.
5. Mix the “butter” sample into the mixing bowl and discard the sample after mixing.
6. Mix the remaining three samples to the AC percentage indicated by the mix design, if there is no mix design then use 5.4% AC.
7. One at a time, place the three samples into the NTO Burn-off Oven at the default burn profile and record the results when completed.
8. Once completed with the NTO Burn-off Oven, wash each sample individually, over the #200 sieve using soapy water.
9. Dry each sample in an oven at  $110 \pm 5^{\circ}\text{C}$ . A sample is considered dry when upon further drying the sample mass does not change by more than 0.1% between two readings.
10. Grade each sample over the indicated sieve series.

11. If performing this test according to AASHTO specifications then Average the first two samples and compare with the “blank” sample. If performing this test according to ASTM specifications, then average all three samples and compare with the “blank” sample.
12. Apply the Aggregate Correction Factor for the Aggregate breakdown if needed. (see AASHTO T-308 for chart).
13. Average the three NTO Burn-off Oven calibrated sample results and compare it with the Actual AC added to each sample.
14. Apply results into the NTO Burn-off Oven.

## **In-House Calibration and Verification Procedure: #20**

Equipment Checked: Dial Gauges

Purpose: This method is used to verify the Dial Gauges.

Equipment Required: Measuring base with guide rod and calibrated dial gauge (see photo below), Latex gloves, Calibrated gauge blocks.

Tolerance: Dial Gauges should be checked every year or if there is reason to question the accuracy.

### Procedure:

1. Before verifying any dial gauges, inspect the measuring equipment to insure that the apparatus is tight. Also inspect the base for any gouges.
2. Install the dial gauge to be checked onto the base and guide rod assembly. Make sure there is no horizontal or vertical movement before proceeding.
3. Wearing latex gloves, check the dial gauges with the following gauge blocks:
  - a. 0.100"
  - b. 0.120"
  - c. 0.150"
  - d. 0.200"
  - e. 0.300"
  - f. 0.500"
  - g. 1.000"
4. If any dimensions are off by more than 0.005", then repeat step #3 to confirm the dimension. If any of the dimensions fail then send the dial gauge in for calibration.

## **In-House Calibration and Verification Procedure: #21**

Equipment Checked: Marshall Molds

Purpose: This method is used to verify the Marshall Molds.

Equipment Required: Cleaning solvent, Cleaning tools (scrapper, towel, etc.) and Calipers.

Tolerance: Marshall Molds should be checked every year or if there is reason to question the accuracy.

### Procedure:

1. Thoroughly clean all parts of the marshall molds.
2. Take and record three measurements at evenly spaced intervals, of each dimension indicated in ASTM D-6926 (written below), with calipers readable to 0.001 in.
  - A. Inside diameter of marshall mold
  - B. Inside diameter of collar lip
  - C. Outside diameter of mold or collar
  - D. Outside diameter of mold lip
  - E. Inside diameter of mold lip, start of the bevel
  - F. Outer diameter of base plate
  - G. Outside diameter of base plate mating surface
  - H. Height of collar
  - I. DOES NOT EXIST
  - J. Height of base plate lip (J1) and height of collar lip (J2)
  - K. Height of mold lip
  - L. Height of mold
  - M. Height of mold bevel
  - N. Total height of mold base
3. Compare measurements obtained with the tolerances given in ASTM D-6926 and AASHTO T-245.

### Note:

- a. AASHTO does not give tolerance on dimensions that ASTM gives tolerances for.
- b. AASHTO requirements on the lip of the mold base are 0.125” and ASTM requirements are 0.235”. This requirement for measurement “J” in ASTM should not affect the test results significantly.



## **In-House Calibration and Verification Procedure: #22**

Equipment Checked: Verify Thermometer Accuracy

Purpose: To insure general use thermometers are accurate and to develop correction charts if they are not accurate. Generally, standard lab thermometers are verified through a temperature differential encountered in the lab, which would be from 15°C to 30°C

Equipment Required: NIST traceable thermometer, thermocouple, or thermistor calibrated yearly, ice, water bath with heating element, circulating impeller or pump.

Tolerance: Thermometers should be verified every year or if there is any reason to question the accuracy of  $\pm 0.2^\circ\text{C}$ .

Procedure:

1. Visually inspect all glass thermometers for chips, fluid separation, and unreadable temperature divisions.
2. Fill the water bath apparatus  $\frac{3}{4}$  full with water and add ice to bring the water temperature to about 15°C. Turn on pump/impeller.
3. Place the NIST traceable thermometer and all of the standard use immersion type thermometers into the bath.
4. Wait at least 10 minutes so that all of the temperature measuring devices acclimate to 15°C. After 10 minutes record each device's temperature.

Note: Make sure that all of the temperature measuring devices ends or leads/probes are at the same elevation in the bath as the temperature may vary on depth/elevation.

5. Raise the bath temperature 3°C at a time and repeat recording the temperatures of each device until the bath temperature is at least 30°C.
6. If necessary produce a calibration curve for the NIST traceable thermometer.
7. Compare all of the lab general use thermometers with the corrected temperatures of the NIST traceable thermometer/thermocouple/thermistor.
8. Produce a calibration curve for each thermometer tested. Dispose of any thermometer if corrections cannot be applied.



### **In-House Calibration and Verification Procedure: #23**

Equipment Checked: Marshall Hammers (Manual & Mechanical)

Purpose: This method is used to verify both mechanical and hand operated marshall hammers.

Equipment Required: 36 in. Ruler readable to 1/64 in, Scale with capacity of at least 20 lbs and readable to 0.01 lbs, and calipers readable to 0.001 in.

Tolerance: Marshall manual hammers should be checked every year or if there is reason to question the accuracy. Marshall mechanical hammer should be checked every three year or if there is reason to question the accuracy.

If you are calibrating/verifying the hammer in accordance with AASHTO, then use tolerances given in AASHTO T-245. If you are calibrating/verifying the hammer in accordance with ASTM, then use tolerances given in ASTM D-6926.

#### Procedure:

1. Clean hammer and remove dirt, tar or other debris build up.
2. Disassemble the top portion of the hammer and remove the hammer weight.
3. Measure and record the mass of the slide hammer. (W)
4. Measure and record three measurements of the face of the tamping end of the hammer. (S)
5. Reassemble the drop hammer
  - a. For the manual hammer
    - i. Adjust the drop height to be  $18.000 \pm 0.025$  in
    - ii. Measure and record the drop height (O-P)
  - b. For the mechanical hammer
    - i. If the drop height is  $18.000 \pm 0.025$  in, then calibration/verification complete.
    - ii. If the drop height is not  $18.000 \pm 0.025$  in, then maintenance is required.

## **In-House Calibration and Verification Procedure: #24**

Equipment Checked: Pressure Meters

Purpose: This method is used to verify and calibrate the concrete pressure meters.

Equipment Required: Assorted screw drivers, wrenches, hammers, chisels, electric drill with wire brushes, muriatic or hydrochloric acid, and vinegar for cleaning. Needed for calibration are: Scale with a 50lb capacity and readable to 0.001lbs, thermometer, glass plates, 5% calibration tube, syringes or squirt bottles, and outer and inner calibration tubes. Paint, markers, and label maker for the completion of the process.

### Procedure:

1. Thoroughly clean each pressure meter with chisels, screwdrivers and wire brushes. Remove all scaling, use acids or vinegar if necessary.
2. Inspect all moving parts to including pumps, petcocks, latches, levers, and gauges to insure proper operation. Replace any inoperable or defective parts, "O" rings and sealing apparatus and check for leaks.
3. Place meter bowl and glass plate on large capacity scale and record weight empty.
4. Fill the meter bowl with 20-30oC tap water and using the glass plate as a screed, insure no air bubbles are trapped with the use of a squirt bottle.
5. Remove any excess water from the outside of the meter bowl and place on the large capacity scale again to record the weight and take a final temperature reading of the water.
6. Insert the inner calibration tube into the top section of the meter making note of the position of the threaded side.
7. Fill the air meter slowly with water through the same valve that has the inner tube attached. Angle the meter as it fills so the opposite valve is elevated and water begins to come out this valve insuring no air resides in the chamber.
8. Pump the air meter up to the calibration number. If one is not known, assume one and the process.
9. Wait 20 seconds for the air in the pressure chamber to cool. Close both valves and release the air from the pressure chamber into the bowl. Observe whether or not the gauge goes to the zero mark from this calibration point.
10. If the assumed calibration number does not go to zero, than repeat the process with another number based on the observations from the previous try.
  - \*Note\* It is very important that all the air is released from the valve without the inner tube and refilled with the valve with the inner tube before performing another try to establish a initial calibration point to the zero mark.

11. Once the calibration zero is determined, refill the meter and insert the outer calibration tube on the same valve that has the inner calibration tube installed on it.
12. Pump up the air pressure chamber and adjust to the calibration mark.
13. Wait 20 seconds to allow the compressed air cool.
14. Place the 5% calibration tube under the outer calibration tube and fill the tube to “level full” by releasing pressure into air meter.
15. Open both valves to release the pressure beginning with the valve without the calibration tubes attached, and let the water drain back into the bowl from the outer tube.
16. Pump up the pressure to the calibration mark determined from the zeroing process.
17. Close both valves and release the pressure from the chamber into the bowl.
18. Observe the reading on the indicator gauge, it should read 5%. If the gauge does not read 5% use the calibration screw to adjust it. If adjustment is needed, repeat the process and verify the 5% mark. It is preferable that the readings at 5% be +/- 0.2%
19. Pump the pressure up again, but do not exceed the max pressure of the gauge.
20. Place the 5% calibration tube under the outer calibration tube and fill the tube to “level full” by releasing pressure into air meter to take an additional 5% out of the air meter bowl.
21. Open both valves to release the pressure beginning with the valve without the calibration tubes attached, and let the water drain back into the bowl from the outer tube.
22. Pump up the pressure to the calibration mark determined from the zeroing process again and let cool.
23. Close both valves and release the pressure from the chamber into the bowl and observe the mark at 10% air. %. If the gauge does not read 10% use the calibration screw to adjust it. If adjustment is needed, repeat the process and verify the 10% mark. It is preferable that the readings at 10% be +/- 0.2%.

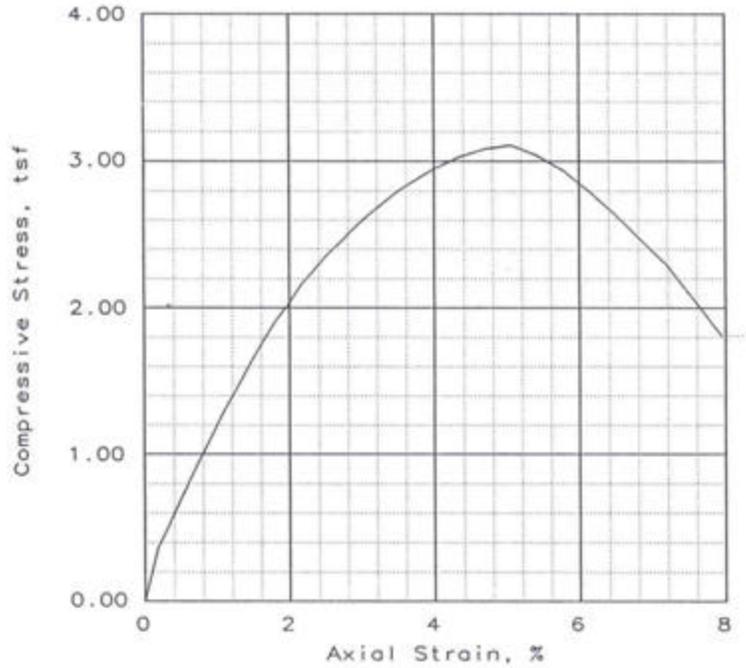
\*calibration of 5% Cal tube\*

24. Using a scale readable to 0.001 lbs, place the 5% air calibration tube with the glass plate and record the weight.
25. Fill the 5% calibration tube up with 20-30oC tap water and using the glass plate as a screed, insure no air bubbles are trapped with the use of a squirt bottle.
26. Record the weight of the tube full of water.
27. Using the provided work sheet and enter all the data collected from both the bowl and calibration tube. The conversion factor for the density of water is pre-determined in the Excel worksheet.
28. With the completed data, compare the volume of the bowl to the calibration tube and determine if the calibration tube meets ASTM C 231 and AASHTO specs.
29. Label the gauge glass of the pressure meter with the determined calibration number. Oil all moving parts and replace any missing tools in kit prior to put back into service.

## **APPENDIX VIII**

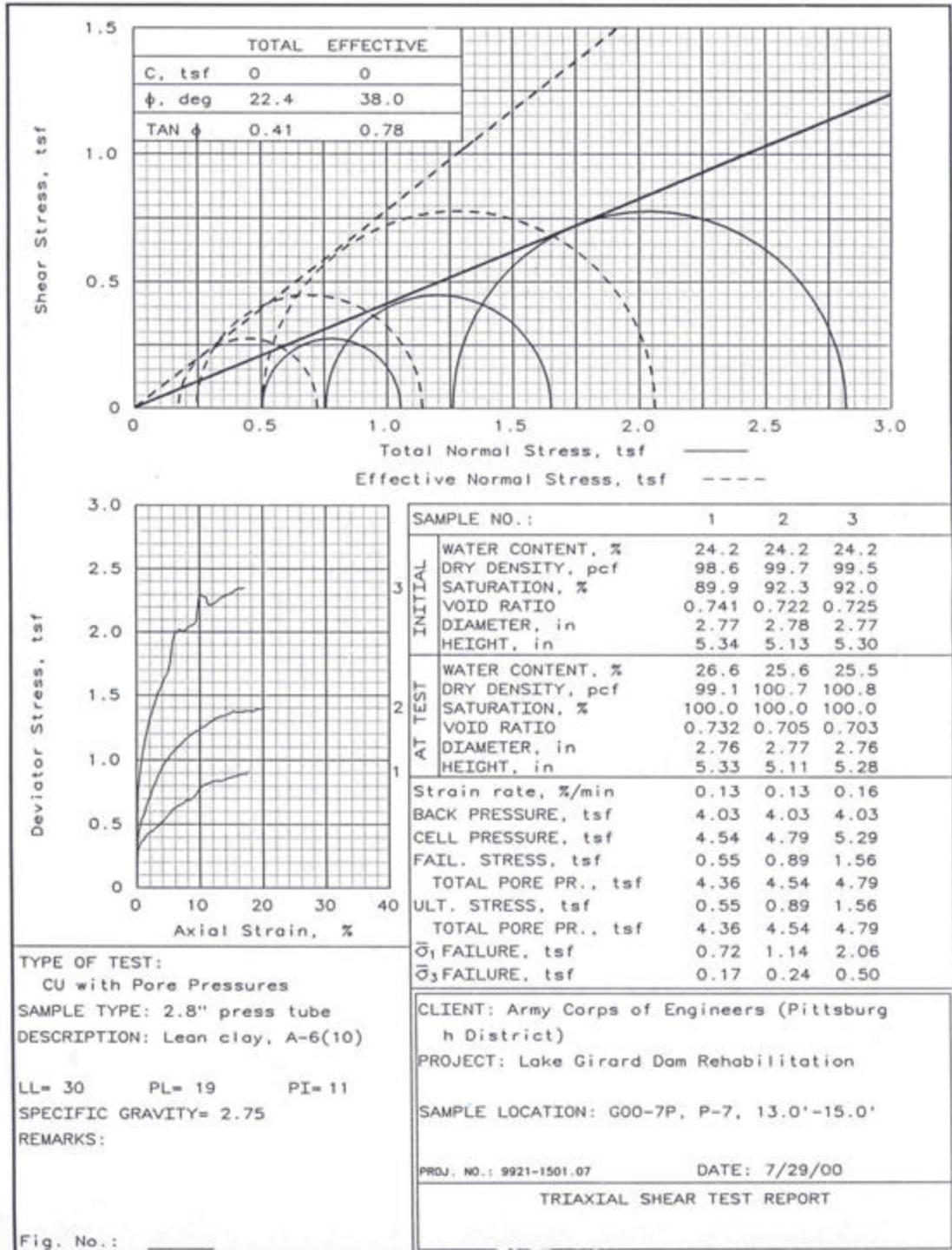
### **Typical Test Report Forms**

### UNCONFINED COMPRESSION TEST



SAMPLE NO.:	1			
Unconfined strength, tsf	3.11			
Undrained shear strength, tsf	1.55			
Failure strain, %	5.1			
Strain rate, %/min	0.88			
Water content, %	13.3			
Wet density, pcf	138.5			
Dry density, pcf	122.3			
Saturation, %	90.5			
Void ratio	0.4041			
Specimen diameter, in	2.87			
Specimen height, in	5.53			
Height/diameter ratio	1.93			
Description: Sandy lean clay, A-4(3)				
LL = 26	PL = 16	PI = 10	GS = 2.75	Type: 2.8" press tube
Project No.: 0021-3061.00		Client: ODOT District 5		
Date: 7/24/00		Project: Hill Diley Road		
Remarks:		Location: RW-B8, P-1, 3.0'-5.0'		
Fig. No.: _____		UNCONFINED COMPRESSION TEST		

**REVISION #8, 01/14/10**



REVISION #8, 01/14/10



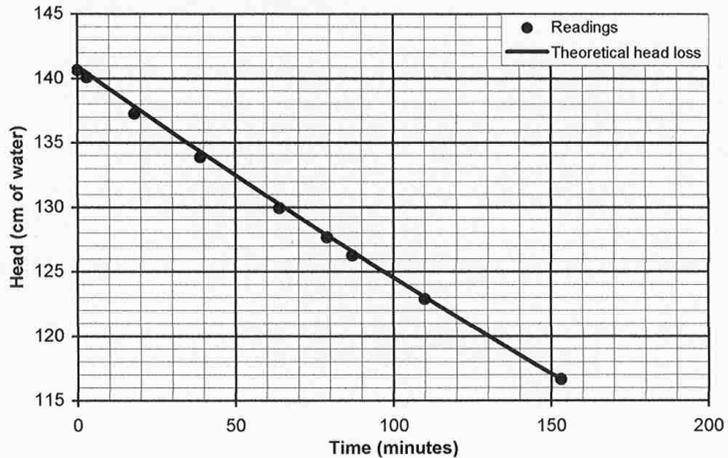
# Permeability Test Report

ASTM D 5084 - Method C

Boring: B-00-10 offset  
 Press Tube: P-1  
 Sample: S-1  
 Depth: 6.5'-8.5'

Liquid Limit	19
Plasticity Index	2
Specific Gravity	2.7

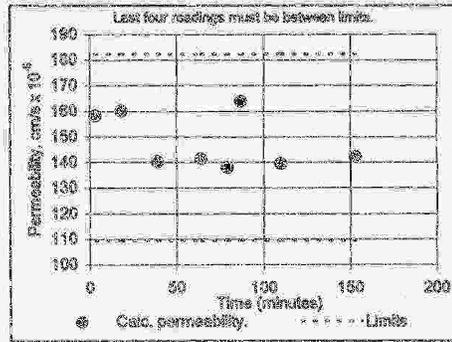
Permeant Liquid	
De-aired, deionized water	
Temp. (°C)	21.0



	Diameter (cm)	Area, A (cm <sup>2</sup> )	Length, L (cm)	Moisture (%)	Dry Density (pcf)	Saturation (%)
Initial	7.109	39.70	8.085	24.2	97.6	89.9
Final	7.109	39.70	8.085	26.9	97.6	100.0

Chamber Pressure (psi)	Backpressure (psi)		Consolidation Stress (psi)		Hydraulic Gradient (-)		
	Inflow	Outflow	Max.	Min.	Max.	Min.	Avg.
60.0	58.0	56.0	4.0	2.0	17.4	14.4	15.8

Date	Time	Buret Readings		Flow Rate	Head, h (cm)	K <sub>arc</sub> (cm/sec)
		Inflow	Outflow			
11/13/00	13:56	0.4	20.0	—	140.6	—
11/13/00	13:59	0.8	19.0	1.00	140.1	1.6E-06
11/13/00	14:14	1.6	18.8	1.00	137.3	1.6E-06
11/13/00	14:35	2.8	17.6	1.00	133.9	1.4E-06
11/13/00	15:00	4.2	16.2	1.00	129.9	1.4E-06
11/13/00	15:15	5.0	15.4	1.00	127.7	1.4E-06
11/13/00	15:23	5.5	14.9	1.00	126.3	1.6E-06
11/13/00	15:46	6.7	13.7	1.00	122.9	1.4E-06
11/13/00	16:29	6.9	11.5	1.00	118.7	1.4E-06



$$k_{20C} = R_T \frac{aL}{2At} \ln \left( \frac{h_1}{h_2} \right) = 1.5E-06 \text{ cm/sec}$$

$a$  - Area of burets (0.71 cm<sup>2</sup>)  
 $t$  - Elapsed time between readings  
 $R_T$  - Temperature correction factor = (0.980)

Remarks:  
 This sample classifies as sandy silt, A-4(0).



Client: Army Corps of Engineers (Pittsburgh District)  
 Project: Belington West Virginia  
 Job No: 0021-4007.03

**REVISION #8, 01/14/10**

**APPENDIX IX**

Approved Subcontractor List

**REVISION #8, 01/14/10**

**Approved Subcontractor List**

Company Name	Address	Contact Information	Work Performed
Calibration Services Inc. (CSI)	300 Buttertown Road Emlention, PA 16373	(800) 793-1870 phone	Calibrations
Accu-Check Instrument Service Inc.	3160 West Fair Avenue Lancaster, OH 43130 www.accucheckinstrument.com	(740) 654-0500 phone (740) 654-3899 fax sales@accucheckinstrument.com	Calibrations
BBC&M Engineering, Inc.	6190 Enterprise Court Dublin, OH 43016 www.bbcm.com	(614) 793-2226 phone (614) 793-2410 fax	Soils Testing
CTL Engineering, Inc.	2860 Fisher Road P.O. Box 44548 Columbus, OH 43204 www.ctleng.com	(614) 276-8123 phone (614) 276-6377 fax	Soils Testing



LF-4.5  
Rev. 1  
12/3/2008

**REVISION #8, 01/14/10**



## **APPENDIX X**

### Revision Logs

**REVISION #8, 01/14/10**

	QUALITY ASSURANCE/QUALITY CONTROL MANUAL DLZ OHIO, INC.	Issue Date:	Rev.:
		05/20/09	6

**APPENDIX IX – Revision Logs**

<b>Revision</b>	<b>Date</b>	<b>Name</b>	<b>Title</b>
1	09/24/08	Barry Wong	Construction Services Manager
2	01/30/09	Barry Wong	Construction Services Manager
3	02/13/09	Barry Wong	Construction Services Manager
4	03/27/09	Barry Wong	Construction Services Manager
5	05/13/09	Barry Wong	Construction Services Manager
6	5/20/09	Jamie North	Construction Services Field Supervisor
7	5/20/09	Jamie North	Construction Services Field Supervisor
8	1/14/2010	Justin Bukey	Senior Technician



**Attachment 2**

**Air Toxics, LTD.**

Laboratory Quality Assurance  
Program, 2009

TO-14A and TO-15 Methods  
Manual, 2009

Reporting Limits for Site Specific  
VOCs, 2010



## LABORATORY QUALITY ASSURANCE PROGRAM

Linda L. Freeman  
CEO, Laboratory Director (Technical Director I)

*Linda L. Freeman* 08/20/09  
Date

Heidi C. Hayes  
Vice President (Technical Director II)

*Heidi Hayes* 8/20/09  
Date

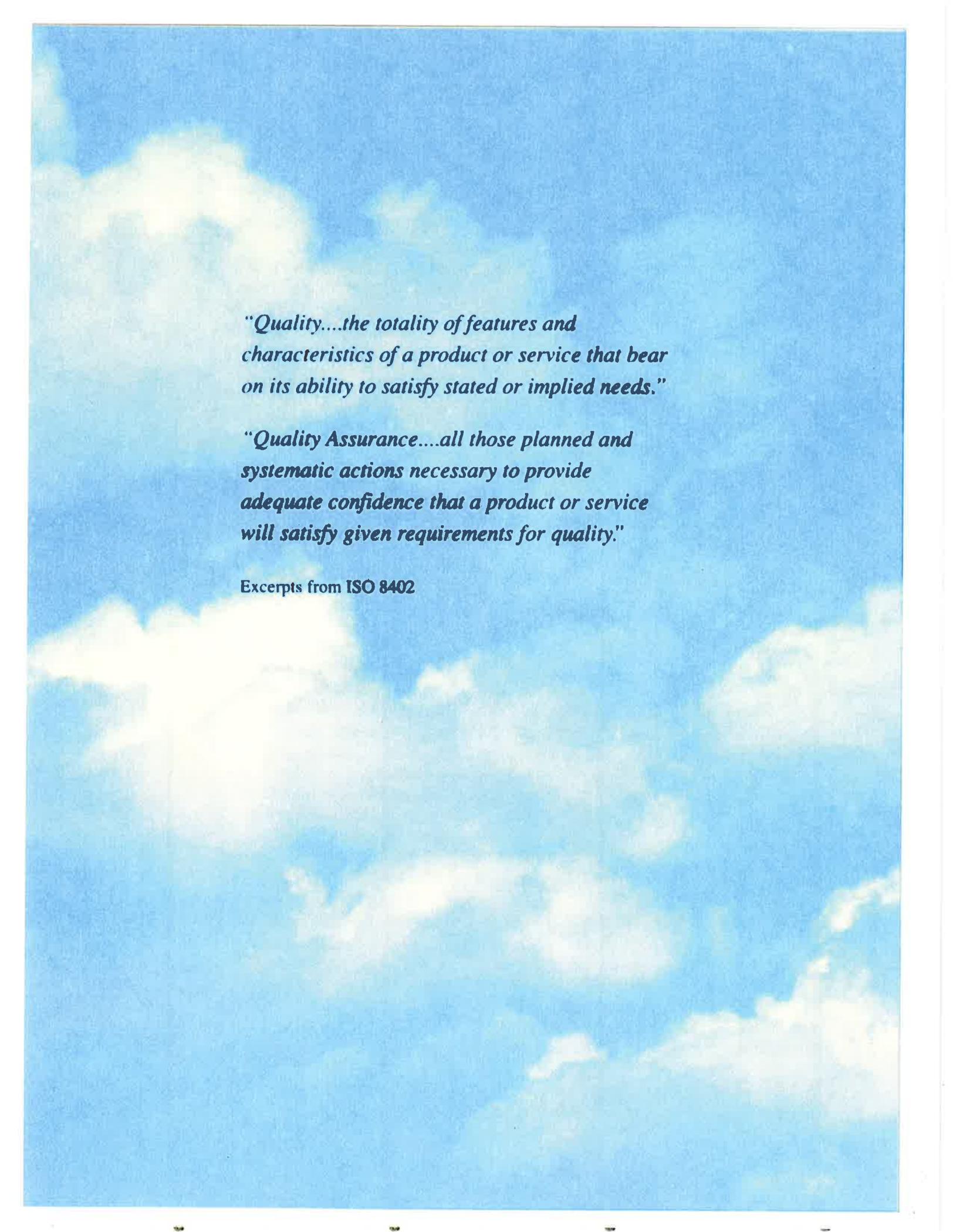
Derek Yokley  
Vice President of Operations

*Derek Yokley* 8/20/09  
Date

Melanie A. Levesque  
Quality Assurance Manager

*Melanie A. Levesque* 08/20/09  
Date

The Quality Assurance Manual is effective as of the date of the signature of the Laboratory Director.



*"Quality...the totality of features and characteristics of a product or service that bear on its ability to satisfy stated or implied needs."*

*"Quality Assurance....all those planned and systematic actions necessary to provide adequate confidence that a product or service will satisfy given requirements for quality."*

Excerpts from ISO 8402

**TABLE OF CONTENTS**

<b>SECTION</b>	<b>PAGE</b>
<b><i>CERTIFICATIONS</i></b>	<b>1-1</b>
<b>1.0 INTRODUCTION</b>	<b>1-2</b>
1.1 QUALITY OBJECTIVE.....	1-3
1.2 QUALITY MANAGEMENT SYSTEM .....	1-3
1.3 INFORMATION MANAGEMENT SYSTEM .....	1-7
<b>2.0 ORGANIZATION</b>	<b>2-1</b>
2.1 STAFF QUALIFICATIONS AND RESPONSIBILITIES .....	2-1
2.2 FACILITIES.....	2-6
2.3 EQUIPMENT AND INSTRUMENTATION .....	2-6
<b>3.0 QUALITY PROGRAM PLAN</b>	<b>3-1</b>
3.1 DOCUMENTING.....	3-1
3.1.1 The Quality Assurance Manual .....	3-1
3.1.2 Standard Operating Procedures/Methods Manual .....	3-1
3.1.3 Revisions to SOPs.....	3-2
3.1.4 Documenting Method Specific Deviations.....	3-3
3.1.5 Documenting Project Specific Deviations .....	3-6
3.2 TRAINING .....	3-7
3.2.1 Team Training.....	3-7
3.2.2 External Training .....	3-8
3.2.3 Quality Training.....	3-8
3.2.4 Health and Safety Training .....	3-8
3.3 ASSESSING ADHERENCE AND COMMUNICATING FINDINGS .....	3-9
3.3.1 Data Review.....	3-9
3.3.2 Corrective Action Program .....	3-10
3.3.3 QA Management Meetings .....	3-13
3.3.4 Conducting Internal Assessments .....	3-13
3.4 COMMUNICATING WITH MANAGEMENT .....	3-13
<b>4.0 QUALITY OBJECTIVES</b>	<b>4-1</b>
4.1 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY .....	4-1
4.1.1 Precision .....	4-1
4.1.2 Accuracy .....	4-1
4.1.3 Representativeness.....	4-1
4.1.4 Completeness .....	4-1
4.1.5 Comparability .....	4-1

**TABLE OF CONTENTS**

<b>SECTION</b>	<b>PAGE</b>
4.2	LIMIT OF DETECTION, LIMIT OF QUANTITATION, AND INSTRUMENT CALIBRATION REQUIREMENTS..... 4-1
4.2.1	Limit of Detection ..... 4-1
4.2.2	Limit of Quantitation..... 4-2
4.2.3	Instrument Calibration..... 4-2
4.2.4	Retention Time Windows..... 4-3
4.3	ELEMENTS OF QUALITY CONTROL..... 4-3
4.3.1	Analytical Batch Definition..... 4-3
4.3.2	Continuing Calibration Verification (CCV)..... 4-3
4.3.3	Laboratory Control Spike (LCS)..... 4-3
4.3.4	Internal Standard (IS) ..... 4-4
4.3.5	Surrogates..... 4-4
4.3.6	Laboratory Blank..... 4-4
4.3.7	Laboratory Duplicate..... 4-5
4.3.8	Matrix Spike..... 4-5
4.3.9	Field QC Samples..... 4-5
4.4	QUALITY CONTROL PROCEDURES..... 4-5
4.4.1	Holding Times..... 4-5
4.4.2	Confirmation ..... 4-5
4.4.3	Standard Materials..... 4-5
4.4.3.1	Liquid Standards ..... 4-6
4.4.3.2	Gas Standards..... 4-6
4.4.3.3	Reagent Water..... 4-6
4.4.4	Expiration Dates of Standards..... 4-7
4.4.4.1	Primary Standards ..... 4-7
4.4.4.2	Secondary Standards ..... 4-7
<b>5.0</b>	<b>SAMPLE HANDLING 5-1</b>
5.1	SAMPLING MEDIA AND PRESERVATION REQUIREMENTS..... 5-1
5.1.1	Sample Containers..... 5-1
5.1.1.1	Summa™ Canisters..... 5-1
5.1.1.2	Sorbent Tubes ..... 5-2
5.1.1.3	Polyurethane Foam (PUF/XAD) Cartridges ..... 5-2
5.1.1.4	DNPH Impinger Solution and Cartridges ..... 5-2
5.2	SAMPLE COLLECTION PROCEDURES - FIELD GUIDELINES ..... 5-3
5.2.1	Information for Canister Sampling..... 5-3
5.2.2	Information for Sorbent Tube Sampling ..... 5-3
5.3	SAMPLE RECEIVING PROCEDURES ..... 5-4
5.3.1	Sample Acceptance Policy ..... 5-4
5.3.2	The Sample Receipt Confirmation ..... 5-5

**TABLE OF CONTENTS**

<b>SECTION</b>		<b>PAGE</b>
5.3.3	The Work Order Folder .....	5-6
5.4	SAMPLE TRACKING PROCEDURES .....	5-6
5.5	INTERNAL SAMPLE CUSTODY AND STORAGE PROCEDURES .....	5-8
5.6	SAMPLE DISPOSAL .....	5-8
5.7	SUBCONTRACTING .....	5-9
<b>6.0</b>	<b>ANALYTICAL METHODS AND PROCEDURES</b>	<b>6-1</b>
6.1	VOST SW-846 5041A .....	6-1
6.2	TO-3 – BTEX AND TPH .....	6-6
6.3	TO-4A/TO-10A – PESTICIDES AND PCBS.....	6-9
6.4	TO-5, TO-11A, METHOD 0011, CARB 430 – ALDEHYDES AND KETONES.....	6-12
6.5	TO-12 – NMOC.....	6-15
6.6	TO-13A AND 8270C – SEMIVOLATILE COMPOUNDS .....	6-17
6.7	TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS.....	6-28
6.8	TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS BY SIM.....	6-34
6.9	ASTM D-1945 – FIXED GASES.....	6-38
6.10	ASTM D-1946 – ATMOSPHERIC GASES .....	6-41
6.11	ASTM D-5504 – SULFUR COMPOUNDS.....	6-44
6.12	TO-17 VOLATILE ORGANIC COMPOUNDS .....	6-47
6.13	PASSIVE SAMPLING – VOLATILE ORGANIC COMPOUNDS.....	6-55
6.14	PM10/TSP.....	6-61
6.15	AIR PHASE PETROLEUM HYDROCARBONS (MA APH).....	6-62
<b>7.0</b>	<b>DATA COLLECTION, REVIEW, REPORTING, AND RECORDS</b>	<b>7-1</b>
7.1	DATA COLLECTION .....	7-1
7.2	DATA REVIEW.....	7-1
7.3	FINAL REPORT PRODUCTION .....	7-2
7.3.1	Automatic Data Transfer System.....	7-2
7.3.2	Manual Data Entry System .....	7-2
7.3.3	Report Compilation.....	7-2
7.4	ELECTRONIC REPORTING AND REVIEW .....	7-4
7.5	eCVP/EDD AND REPORTING IN ADOBE FORMAT OR DISKETTE.....	7-4
7.6	RECORDS OF METHOD CAPABILITY .....	7-7
7.7	RECORD STORAGE .....	7-7
7.8	CONFIDENTIALITY OF DATA .....	7-9

**TABLE OF CONTENTS**

<b>SECTION</b>	<b>PAGE</b>
<b>8.0 ESTABLISHING ACCEPTANCE CRITERIA</b>	<b>8-1</b>
8.1 CONTROL CHART PROGRAM .....	8-1
8.2 ESTABLISHING CONTROL LIMITS .....	8-1
8.3 INTERPRETING CONTROL LIMITS .....	8-1
8.4 MEASUREMENT UNCERTAINTY .....	8-1
<b>9.0 PREVENTATIVE MAINTENANCE</b>	<b>9-1</b>
9.1 ROUTINE MAINTENANCE .....	9-1
9.2 SERVICE CONTRACTS .....	9-1
9.3 SPARE PARTS INVENTORY .....	9-1
9.4 CONTROL OF MISCELLANEOUS MONITORING, MEASURING, TESTING, AND DATA COLLECTION EQUIPMENT .....	9-2
9.4.1 Analytical Balances and Weight Sets .....	9-2
9.4.2 Pressure Gauges .....	9-2
9.4.3 Fume Hood Testing Device .....	9-2
9.4.4 Thermometers .....	9-3
9.4.4.1 Reference Thermometers .....	9-3
9.4.4.2 Working Liquid-Filled Thermometers .....	9-3
9.4.4.3 Oven and IS Station Thermometers .....	9-3
9.4.4.4 Non-Contact Thermometers .....	9-4
9.4.5 Temperature/Humidity Recorders .....	9-4
9.4.6 Flow Meters .....	9-4
9.4.7 Mass Flow Controllers .....	9-4
9.4.8 Mechanical Volumetric Devices .....	9-5
9.4.9 Oven Vacuum Gauges .....	9-5
<b>10.0 PROFICIENCY TESTING PROGRAM</b>	<b>10-1</b>
10.1 NELAP PT TESTING PROGRAM .....	10-1
10.2 EXTERNAL (NON-NELAP) PT SAMPLES .....	10-2
<b>11.0 MANAGEMENT OF COMPUTER AND SOFTWARE SYSTEMS</b>	<b>11-1</b>
11.1 SECURITY .....	11-1
11.2 BACK UP AND STORAGE OF DATA .....	11-1
11.3 SOFTWARE AND ELECTRONIC DATA VALIDATION .....	11-1
11.3.1 Stage I – Alpha Testing .....	11-2
11.3.2 Stage II – Beta Testing .....	11-2
11.3.3 Stage III – Implementation .....	11-3
11.3.4 Commercial Software Data Validation .....	11-3

**TABLE OF CONTENTS**

<b>SECTION</b>	<b>PAGE</b>
<b>12.0 CONTROL OF PURCHASED ITEMS AND EXTERNAL SERVICES</b>	<b>12-1</b>
<b>13.0 THE PROJECT MANAGEMENT SYSTEM</b>	<b>13-1</b>
13.1 REVIEW OF PROJECT SPECIFIC DOCUMENTS .....	13-1
13.2 NEGOTIATIONS AND VARIANCE REQUESTS.....	13-5
13.3 DOCUMENTATION OF PROJECT REQUIREMENTS.....	13-5
13.4 DOCUMENTING CLIENT DISCUSSIONS .....	13-5
13.5 DOCUMENTING CLIENT CONCERNS.....	13-5
13.6 SCHEDULING SAMPLING MEDIA.....	13-6
13.7 TRACKING SAMPLE ANALYSIS AND REPORTING.....	13-6
13.8 PROJECT FOLLOW -UP .....	13-6
<b>14.0 DATA INTEGRITY PROCEDURES</b>	<b>14-1</b>
14.1 TRAINING.....	14-1
14.2 PERIODIC MONITORING .....	14-2
14.3 MECHANISMS FOR REPORTING INFRACTIONS .....	14-2

---

**APPENDICES**

- A. DEFINITIONS & TERMS
- B. LIST OF STANDARD OPERATING PROCEDURES (SOPs)

**REFERENCES**

**This page left blank intentionally**

This Quality Manual was designed to meet 2003 NELAP (National Environmental Laboratory Accreditation Program) standards as well as the Department of Defense Quality Systems Manual Version 4.1 and supports assessment programs and/or certifications with the following agencies:

<b>Certifying Agency</b>	<b>ATL Certificate #</b>	<b>Basis of Certification/Approval</b>	<b>Location of Certificate and Parameter List</b>
Arizona DHS	AZ0719	Onsite assessment (annual), LQAP and SOP	Laboratory internal network: O:\QA\Certifications
California DPH (Primary NELAP)	02110CA	Onsite assessment (biennial) LQAP, SOP and WP PTs	Laboratory internal network: O:\QA\Certifications
Florida DOH (Primary NELAP)	E87680	Onsite assessment (biennial) LQAP and SOP Review	Laboratory internal network: O:\QA\Certifications
Louisiana DEQ	02089	LQAP, SOP Review, WP PTs, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
New York State DOH	11291	LQAP, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Oregon DHS	CA200012-001	Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Texas CEQ	T104704434-08-TX	LQAP, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
State of Utah DOH	AIR	LQAP, WP PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
Washington DOE	C2067	PT, Secondary NELAP	Laboratory internal network: O:\QA\Certifications
U.S. Navy NFESC/IR/QA	NA	DOD QSM for Environmental Laboratories v.3/ Recognition of NELAP Accreditation	Laboratory internal network: O:\QA\Certifications
Pennsylvania State Dept. Health	68-690		Laboratory internal network: O:\QA\Certifications

## MANAGEMENT QUALITY POLICY STATEMENT

At ATL, we strive to be the **BEST** in everything that we do. Our very existence is based on our continued ability to provide innovative, dependable, and cost effective environmental services to our clients. We **CARE** about our clients as well as our co-workers and manage our daily activities to build relationships based on mutual **TRUST, HONESTY, and RESPECT**. We are **LEADERS** in our field and accept the risks associated with building new frontiers in our professional lives. Our strength comes from our **TEAMS** for through them we can achieve our goals. Our business is guided by four key principles: 1) Proving unmatched data integrity; 2) Establishing long term relationships; 3) Delivering quality client service; and 4) Exceeding client expectations. This Quality Assurance Manual defines and documents the core systems surrounding good professional as well as laboratory best practices for all staff in accordance to NELAC standards. As such, all laboratory staff are required to familiarize themselves with the quality documentation and implement these policies and procedures in their work. The management signatures below represent our commitment to the NELAC standards and the Department of Defense Quality Systems Manual Version 4.1 as well as and our commitment continually define, assess, and improve the quality systems, which provide the basic infrastructure in support of these goals.

## 1.0 INTRODUCTION

The Air Toxics Limited (ATL) Quality Manual describes the Quality Assurance (QA) program and Quality Control (QC) procedures used to ensure that data of known and documented quality are produced. It is designed to be used as a manual that outlines the process by which we ensure that the customer expectations are met, and hence, the quality goal is met. *ISO/IEC Guide 17025-General Requirements for Competence of Calibration and Testing Laboratories* are incorporated wherever possible, however the primary guidance document is *Chapter 5: Quality Systems* as published in the June 5, 2003 NELAP Standard and the Department of Defense Quality Systems Manual Version 4.1.

The Quality Manual contains a discussion of the following topics:

**Introduction:** The quality objective is discussed along with management and information systems in support of the objective.

**Organization:** Staff qualifications and responsibilities, management organization, laboratory facilities, and equipment are detailed in this section.

**Quality Assurance Program:** This section deals with project management, standard operating procedures, staff members' training, evaluation and documentation of adherence to quality assurance and quality control requirements, corrective action system, and health and safety.

**Quality Objectives:** This section explains the quality control parameters and procedures, procedures to establish limits of detection and quantitation and perform calibrations, traceability, and preparation of standards.

**Sample Handling:** Sampling containers, preservation and Chain-of-Custody requirements, sample receiving and tracking

procedures, internal custody, storage and disposal are discussed.

**Analytical Methods and Procedures:** In this section, a brief method description is given for all analytical procedures carried out at ATL. The limit of quantitation concentrations, quality control acceptance criteria and method modifications are provided as well.

**Data Review and Reporting:** This section explains the procedures involved in data collection/reduction, data review, and final report production. Electronic data production, data flagging, and data storage are also discussed in this section.

**Establishing Acceptance Criteria:** The control chart program is outlined in this section along with generating and evaluating in-house statistical limits.

**Preventative Maintenance:** Routine maintenance, service contracts, and control of miscellaneous monitoring equipment are explained briefly.

**Assessments and PT Samples:** A brief explanation of internal and external assessments programs and NELAP PT samples program is provided.

**Computer and Software Systems:** This section of the quality manual deals with the management of computer and software systems. Data storage, back-up routines, and internal software validation efforts are included.

**Control of Purchased Items:** Control of purchased items and external services as well as the purchase requisition system are outlined in this section.

**Project Management System:** This part of the quality manual gives a brief description of steps to ensure that the customer expectations are met once the project is undertaken.

## 1.1 QUALITY OBJECTIVE

Air Toxics Limited is committed to producing data that meet or exceed the client's measurement needs. Customer satisfaction is the motivating force behind most of the ATL processes. An underlying network of systems designed to define, document, and process each individual customer's need supports this primary objective. This systems network includes Sales, Project Management, IT, Laboratory Production, Support Services, Technical Services, Quality Assurance, and Finance. Each of these operational areas is organized around an empowered work team accountable for delivering an automated, on time and defensible result.

We believe the ultimate responsibility for quality resides at the team level. Every team member has the responsibility and authority to suspend a process if it appears that the quality objectives are not being met. Analytical team members are informed of the quality objectives via documented Standard Operation Procedures (SOPs) and project related information systems (ATL's Project Profiles and Project Requirement tables). Team members work closely with the Project Management and QA Departments to ensure that the quality objectives are met.

## 1.2 QUALITY MANAGEMENT SYSTEM

The role of the ATL management team is to design, implement, maintain and audit the quality systems. Quality systems are designed and documented on each functional team through Standard Operating Procedures. Once designed, the management team ensures through ongoing daily activity that adequate funding, equipment, staffing and training exist to support the success and effectiveness of the quality systems. Management helps ensure that the quality objective is met by establishing a continuous and reiterative program of audit review and process improvement. Establishing goals at the team

and individual levels that support the objectives of the quality systems is an essential management activity as well providing adequate individual accountability measures and feedback. The primary role of the management team is to establish strategic performance goals at the corporate and team levels and to produce quantifiable measures of performance against these goals (e.g., customer satisfaction index, sales quotas, report turn around time, net profit, days to complete corrective actions, etc.). The success or failure of every relevant quality system is the ultimate responsibility of management. The management team consists of Business Directors, Department Managers and Team Leaders.

**Quality Assurance Management:** The role of the ATL Quality Assurance team is to help ensure that the systems described above are designed, documented, and operating in accordance with the quality objectives. This is accomplished via coordination and dissemination of internal and external assessment information, review of SOPs to document variances taken to published methods, monitoring of the Quality Manual to assure consistency with actual practices, maintenance of an ongoing Corrective Action Program with quarterly reports to management, and a leadership role in employee training programs. A secondary function of the QA team deals with data review and other quality control related programs.

The QA team is free from any commercial, financial, or production pressures when making assessments or decisions regarding the quality of work produced or effectiveness of the quality systems. The Quality Assurance Manager reports directly to the Vice President/Technical Director II in order to maintain independence from business operating units and facilitate communications regarding quality related issues.

Communication between the QA team and other management teams occurs on a regular basis via bi-weekly status meetings. Information regarding outstanding corrective action items, upcoming assessments, assessment results or general observations are brought up and documented via a database of agenda notes. The quality assurance database along with the ATLAS database are used to compile a 'Quarterly Quality Assurance Status Report', which is distributed to the entire management team for review.

**Sales and Project Management:** The role of the ATL Sales and Project Management teams is to effectively document and communicate the needs of the customer. These teams represent the customer in all matters and serve as a liaison between the customer and the Technical Services, Laboratory, Support Services, Finance, and Quality Assurance areas. The ATL Sales and Project Management teams work with laboratory management to ensure that client needs are matched by laboratory resources. Strong communication linkages exist between the Laboratory Department Managers, Team Leader and the ATL Sales and Project Management teams. Information regarding customer needs flows into all ATL systems via these two teams. Interactions may be as complex as Quality Assurance Project Plan (QAPP), contract or Scope of Work (SOW) review or as simple as processing shipments of canisters and other sampling media. Project specifics are documented and stored via an interactive database that assigns a unique identifier for every reference. Every contact with the customer is documented in the database by the Project Management team so that management and laboratory technical staff can make decisions based on electronic sorted one touch information.

**Sample Receiving:** The goal of the department is to enable every sample to be received and processed into a unique laboratory Work Order within 24 hours of

sample receipt. Sample non-conformities are communicated to the clients in the same time frame. Custody information relating to sample receipt, a copy of the sample receipt summary, and the compound list and reporting limits for the requested analysis is e-mailed or faxed to the client for review and comments.

**Laboratory Management:** Laboratory management is divided into work teams equipped with necessary resources to complete the sample analysis, review, data reporting and creation of all electronic data packages, which include e-mail, EDD and eCVP on CD-ROM. The laboratory work teams are responsible for verifying the quality of electronic deliverables by reviewing a percentage of the product. In this way, team members are easily able to accept the control and accountability for quality. The Support Services team is responsible for cleaning, assembling, coordinating media certification and shipping all sampling media. The primary responsibility of the Team Leader and/or Department Manager is to monitor customer needs versus resource availability. Staff and equipment management are carefully balanced with customer needs. The goal for each team is to deliver defensible data within the time frame promised to the client. The Department Managers or designated personnel review daily sample receipt work lists to determine that the laboratory has adequate resources to perform the work. In those cases where either the technical or sample capacity demands cannot be met, the Department Manager works with the Project Manager and the client to provide a solution via inside resource re-allocation or outside subcontracting. The ATLAS laboratory automation system creates and tracks special analytical lists, deliverables, or Turn Around Time (TAT) requests which are automated via customized linkages (work tools) into the centralized Structured Query Language (SQL) database. Performance measurements against the goal are routinely monitored using the same SQL database. Performance and quality related information is

shared with team members during team meetings. Project or client related information resides both in the project management module and in sample tracking modules, reducing the need for relying on verbal communication of project specifics to the team. Laboratory management and personnel are free from any commercial, financial, or production pressures when making technical judgments or decisions regarding the quality of work produced. The Team Leaders report to a Department Manager who reports to the Vice President of Operations.

The remaining team positions are divided into four levels:

**1) Senior Scientist (Lab Personnel) or Senior Associate (non-Lab Personnel):**

This is the highest level professional position reporting to a Director or Department Manager. The Senior Scientist or Senior Associate works independently at a company wide level. In addition to all of the responsibilities of the Scientist described below, the Senior Scientist or Senior Associate is recognized within the company as an expert in his/her field. He or she is often asked to work outside of the team whenever the need arises, and is able to demonstrate above average leadership skills. Senior Scientists or Senior Associates are responsible for method development activities and take a lead role in proprietary software and hardware design and testing. High profile projects or client relations, including more intricate analyses and data interpretations, are assigned to a Senior Scientist to oversee. The Senior Scientist or Senior Associate maintains knowledge at the level of Masters Degree or equivalent with a minimum of 5 years of analytical environmental experience.

**Scientist (Lab Personnel) or Associate (Non-Lab Personnel):**

The Scientist or Associate works independently at the team level. A Scientist or

Associate demonstrates a high level of skill, judgment, problem solving ability, and is able to independently perform troubleshooting. The responsibilities of a Scientist/Associate include: scheduling of work, providing routine as well as non-routine bench level activities in a highly efficient manner, writing SOPs, reviewing data, performing non-routine instrument maintenance and troubleshooting, and representing the team during internal/external assessments. Individuals in this position play a lead role in monitoring health and safety on the team, and acting as a resource or trainer. A Scientist or Associate must have a Bachelor's Degree and a minimum of three years of analytical environmental experience.

**Analyst (Lab Personnel and Non-Lab Personnel):**

The Analyst works under the direct supervision of a Scientist, Senior Scientist, and/or Department Manager at all times. He/she follows a specific formal training program to learn the necessary skills required of the position and demonstrates the ability to recognize problems and to seek assistance. The primary responsibility of an Analyst is to follow written laboratory SOPs in an efficient and well-informed manner. The Analyst performs routine maintenance on the equipment, prepares standards, and performs all relevant bench level activities. The minimum qualification for a laboratory analyst is a Bachelor's Degree.

Each team has a mix of Scientists and Analysts. Each Department Manager coordinates the activities of the respective team, serves as a resource to the Analysts and Scientists, communicates the corporate objectives to the team, and monitors team progress against the quality objective, which is customer satisfaction. A Scientist may be assigned as 'team lead' in a subset of team activities. As 'team lead' the Scientist is responsible for all the technical activities of the assigned area, oversees both the quality

and quantity of work produced, and serves as resource for the Analysts. The Analyst performs all of the routine activities and quality checks (i.e., makes sure the customer expectations are met).

Every team member is empowered to make sure that the customer expectations are met, is trained in the elements of the quality process, and has the responsibility and authority to stop or suspend a process when the quality objective is in jeopardy.

**Technician** (Lab Personnel and Non-Lab Personnel):

The Technician works directly under the supervision of a more experienced team member, Team Leader and/or Manager at all times. He/she is responsible for meeting the team's production and quality goals. The Technician performs a variety of tasks in whatever area of the laboratory they are assigned. For example, the Technician's responsibilities may include washing and solvent rinsing glassware, cleaning and preparing media to ship to clients, pressurizing and screening samples, logging samples, assist in preparing standards and other duties as assigned. He/she is also expected to communicate issues to a team Scientist and/or Team Leader. The minimum qualification for a Technician is a High School Diploma.

**Information Technology:**

The Information Technology (IT) team is responsible for the design and maintenance of the SQL server based data system. Its primary goal is to ensure that customer satisfaction is achieved by the way information is transferred, processed, or queried. This includes systems relating to telephone service, e-mail service, Internet access, project management, data acquisition, assessment trails, data security, and automated data reporting linkages. The group consists of the IT Manager, and one full time programmer.

Around-the-clock, system support is achieved via a combination of in-house and contract support. Additional programmers are hired on a project specific basis. All of the ATL information systems are designed, coded, and tested in house and as such, are proprietary in nature. The IT Manager reports to the President.

**Financial Management:** The quality systems rely on bottom line profitability to provide strength to the framework that produces quality results. The ATL Finance team is responsible for monitoring the profitability of all operations. Customer satisfaction goals are built into budgeting, purchasing, invoicing, employee compensation and benefits programs, collections, contracts, insurance, and banking. The primary goal of the team is help ensure bottom line profitability while achieving the quality objective, which is customer satisfaction. The group consists of a Controller, a Finance Associate, a Credit & Collection Associate and an Accounts Payable/Purchasing Associate. The Finance Manager reports to the President.

**Data Integrity Procedures:** Since a commitment to data integrity is a vital component for credibility of our core product, Air Toxics Limited cannot function as a business entity without a clear definition of ethical expectations for all employees. Integrity is defined as the ability to discern right from wrong, and the commitment to do what is right, good and proper. Data integrity procedures relating to generation of analytical reports are built into the systems via the operational SOPs, which describe appropriate practices. Additional systems and training programs that safeguard strict adherence to the SOPs ultimately ensure that data integrity procedures are employed. Intentional fraud will be grounds for severe reprimand and/or termination of complicit employees. In addition, employees who witness or are otherwise aware of data integrity violations, even if they are not a party to such acts, are

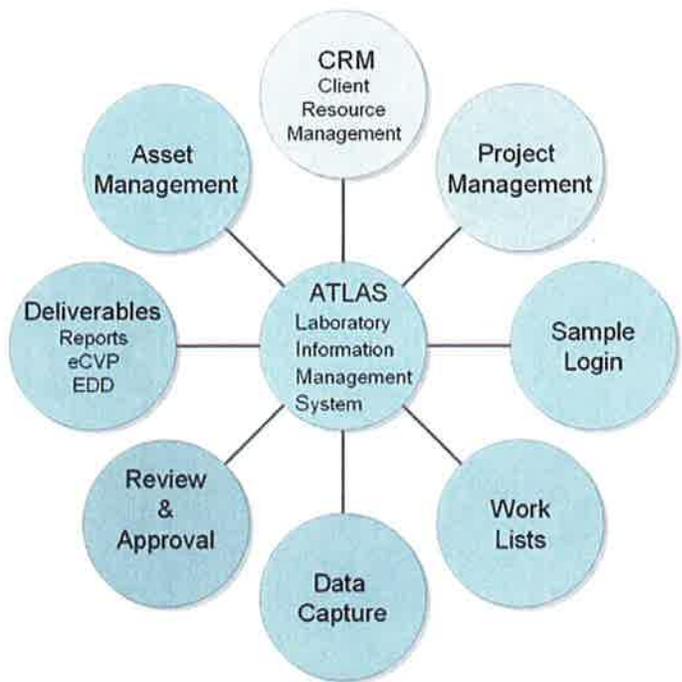
expected to immediately report these lapses to their Team Leader, their Department Manager or to a member of the Board of Directors.

Data integrity training is conducted within a variety of frameworks and is mandatory for all Air Toxics Limited employees. New employees read both the LQAP and the Employee Handbook to properly orient ethical expectations. In addition, within two months of date of hire there is basic training provided by the Quality Assurance Department to familiarize new employees with principles of documentation to pre-empt practices that would call into question the data integrity procedures of the laboratory. The Chief Executive Officer of Air Toxics conducts a Standards of Conduct presentation that defines data integrity expectations, potential penalties and consequences for lapses of integrity for new employees as they join the company. In addition, the CEO conducts a yearly ongoing Ethics and Integrity Training II for the remainder of the employees. The Inappropriate Lab Practices Class (also done on a yearly basis) defines allowable parameters for the lab to assure defensibility and to define illegal practices. The purpose of all training is to provide specific examples of data integrity expectations that are relevant to actual job functions. Employees document the training in their training records (see Section 3.2.1).

### **1.3 INFORMATION MANAGEMENT SYSTEM**

Information is stored in the Air Toxics Laboratory Automation System (ATLAS) databases using ATL designed hardware and proprietary software. This in-house Laboratory Information Management System (LIMS) is an evolving development project designed to find more efficient means to meet the customer needs. Each client contact (telephone call, quote, shipping request, or inquiry) is stored in a database, which can be queried for sample log-in, project backlogs, project TAT or revenue statistics.

Some modules are designed to track non-traditional information such as the sample history of individual canisters, number of reports completed per analyst per shift, and overdue work by reason code. These types of information directly affect the ability of the management team to provide quality process improvements. Some non-traditional calculations such as the boiling point distribution of a hydrocarbon background, EPA rounding, and percent difference calculations have been made available at the bench. This type of information directly affects the ability of the individual employee to meet the quality objectives.



**This page left blank intentionally.**

## **2.0 ORGANIZATION**

### **2.1 STAFF QUALIFICATIONS AND RESPONSIBILITIES**

ATL's management organization includes the Board of Directors comprising four core areas: Operations, Finance, IT, and Sales. In addition there are Department Managers, Area Managers and Team Leaders. Each operating area is either lead by a Department Manager or a Team Leader. Due to the size and complexity of the main laboratory, Department Managers and Team Leaders are required. Most Managers and Team Leaders report to a member of the Board of Directors. In the absence of a member of the management team responsibilities are fulfilled as follows: If the President is absent, the Vice President/Technical Director II may fulfill the responsibilities as President. In addition, if the primary Technical Director is absent the second Technical Director will fulfill the responsibilities. If the QA Manager is absent, the Technical Directors may fulfill QA responsibilities. In the absence of a Manager or Team Leader, one of the Directors will name an interim successor.

#### **LINDA L. FREEMAN CHIEF EXECUTIVE OFFICER AND LABORATORY (TECHNICAL) DIRECTOR (I)**

Ms. Linda L. Freeman is the Technical Director and the Chief Executive Officer of ATL providing leadership that ensures the founding mission and core values of the company are put into practice. Ms. Freeman leads programs relating to the development of long range strategy, quality systems, and financial infrastructure. As Technical Director (I), her responsibilities include: the administrative review of laboratory operations and qualifications for the technical positions, ensuring and documenting initial and ongoing proficiency, and oversight of the Quality systems. She holds a Bachelor's Degree from Boston College and a Master's Degree in

Chemistry from the University of Wisconsin-Madison. Ms. Freeman has over 22 years of combined environmental experience and 19 years of laboratory business management experience.

#### **BRAD MOSAKOWSKI PRESIDENT**

Mr. Mosakowski is the President of Air Toxics Ltd. and represents the partnership in all matters. Mr. Mosakowski provides day-to-day leadership and management of programs for overseeing the processes and resources necessary for establishing long-range service objectives, plans and policies, in cooperation with the CEO and Board of Managers. He is responsible for the measurement and effectiveness of both internal and external processes by providing accurate and timely feedback on the operating condition of the company. In addition, Mr. Mosakowski also directs the definition and operation of the laboratory production by fostering a success-oriented and accountable environment within the company. A critical component of which is his ability to motivate and lead a high performance management team capable of meeting both customer service and bottom line financial objectives. Mr. Mosakowski has over 19 years of combined environmental laboratory experience.

#### **HEIDI C. HAYES VICE PRESIDENT AND TECHNICAL DIRECTOR (II)**

Ms. Heidi C. Hayes is the Vice President and Technical Director (II) of ATL. Ms. Hayes provides the technical leadership, management and vision necessary to ensure the company has the proper operational controls, quality systems, administrative procedures and human resource management in place to meet customer need and quality objectives. In her role as Technical Director, she manages the method development efforts and oversees the design, development, and implementation of new products and technical innovations. Ms.

Hayes is also the key technical interface between laboratory services and major clients and regulators. With over 15 years of environmental laboratory experience and 12 years of management experience, Ms. Hayes holds a Bachelor's Degree in Chemistry and Mathematics from Luther College and a Master's Degree in Chemistry from the Colorado School of Mines.

**DEREK YOKLEY**  
**VICE PRESIDENT OF OPERATIONS**

Mr. Yokley serves as the Vice President of Operations for Air Toxics Ltd. He is responsible for overall operational leadership for laboratory and the supportive services. Mr. Yokley comes to Air Toxics with 14 years manufacturing/quality assurance leadership experience. He has spent 10 of those years applying the Toyota Production System (Lean Manufacturing) in various industries both as an employee and as a consultant—most recently with General Electric. Mr. Yokley serves as the Lean Champion and is guiding the company through its Lean transformation and continued journey towards operational excellence. He holds a Bachelor's degree in English from California State University Sacramento. He is a Senior Member of the American Society for Quality and has earned the certification Manager of Quality/Organizational Excellence.

**MELANIE LEVESQUE**  
**QUALITY ASSURANCE MANAGER**

Ms. Melanie Levesque develops and supervises programs intended to ensure that the laboratory is producing data of known and acceptable quality. Ms. Levesque oversees QC activities including various independent checks of laboratory systems, maintenance of the Laboratory Quality Assurance Manual, SOP generation, and corrective action procedures, as well as monitoring laboratory certification programs. Ms. Levesque has documented training in the approved methods and can verify that the laboratory is following

SOPs. Ms. Levesque maintains independence from the operations by not engaging in production activities and reports directly to the Vice President/Technical Director II. The QA Department conducts a yearly independent audit of the quality systems and methods criteria, and notifies laboratory directors of deficiencies via a written report. Ms. Levesque holds a Bachelor's degree in Chemistry and a Master of Science degree in Analytical Chemistry both from Rochester Institute of Technology, followed by ten years of environmental laboratory experience. Ms. Levesque has worked in a variety of positions including HPLC chemist, GC/MS chemist, and laboratory supervisor.

**NATHAN SHAFER**  
**LABORATORY DEPARTMENT MANAGER**

Mr. Nathan Shafer is the Department Manager for the Volatile Organic Compound (VOC) GC/MS analysis group. This department is responsible for all analyses via methods TO-14A/15, VOST methods 0030 and 0031, TO-17, and all VOC pptv work in the area of vapor intrusion. Mr. Shafer is responsible for managing and overseeing all processes and resources involved in the daily operations of the VOC department. In addition, he provides technical support to project managers, sales, and the department; he is also responsible for coaching and training team members, data review, scheduling, and conferencing. Mr. Shafer has been employed by Air Toxics since 1997 and has 11 years of environmental laboratory experience. His experience comes from roles such as GC/MS chemist, laboratory supervisor, and project development chemist. Mr. Shafer holds a dual degree from Claremont McKenna College in the fields of chemistry and psychology.

**SEPIDEH SAEED**  
**LABORATORY DEPARTMENT MANAGER**

Ms. Sepideh Saeed is the Department Manager for the GC, HPLC and GC/MS semi volatiles

analysis, which includes EPA Method TO-3/TO-12, ASTM D-1945/1946, TO-14A/TO-15 5&20/Soil Gas, SW8260B, Extractions, ASTM D-5504, TO-13A, TO-5/CARB430, TO-11, Method 0011, PM10, TSP, NIOSH 5515, Siloxanes, Pesticide and PCB Analytical Group. She is responsible for managing and overseeing all processes resources involved in the daily operations of SVOC department. Ms. Saeed also provides technical support to project managers, sales, and the department; she is also responsible for managing and coaching team members, data review and conferencing. Ms. Saeed has been employed at Air Toxics since 1998 and has over 16 years of environmental laboratory experience. Her experience comes from roles such as a GC, HPLC, GC/MS and extraction chemist and laboratory supervisor. Ms. Saeed has a B.S. Degree in Biochemistry from University of California, Davis.

**NOOR KHAN**  
**TECHNICAL SERVICES DEPARTMENT**  
**MANAGER**

Mr. Noor Khan is the Department Manager for the technical services group. This department is responsible for the maintenance of all instrumentation and the implementation of new technologies to maximize laboratory performance. Mr. Khan is responsible for managing and overseeing all processes and resources involved in the daily operations of the technical services department. In addition, he ensures prompt response to instrument issues, manages the troubleshooting processes and ensures the outcome meets the user's needs while operational and quality goals are met; he also works to develop staff through technical support and technical training. Mr. Khan has been employed by Air Toxics since 1998 and has 11 years of environmental laboratory experience. His experience comes from roles such as GC/MS chemist and senior scientist. Mr. Khan holds a Master of Science degree in chemistry from University of Karachi, Pakistan and a Master of Science

degree in Analytical Chemistry from University of Texas San Antonio

**MICHAEL GRIFFIN**  
**SALES & MARKETING MANAGER**

Mr. Griffin is the Sales & Marketing Manager of Air Toxics, Ltd. He is responsible for establishing and leading the sales and marketing efforts through the development and implementation of strategies to generate profitable sales and growth of the company. He also provides the leadership, management and vision necessary to the project management team to exceed client expectations. Mr. Griffin has 20 years of technical sales experience and 10 years in Sales and Operations Management. Mr. Griffin holds a bachelors degree in Business Administration with a concentration in International Business Management from California Polytechnic State University San Luis Obispo.

**KEN ZELENY**  
**INFORMATION TECHNOLOGY MANAGER**

Mr. Zeleny is the Information Technology Manager for the IT Group. His responsibilities include database management, software development and network management. Mr. Zeleny has over 19 years experience with computer and technology functions in both large and small organizations. His experience also includes 5 years as a Sr. Systems Architect and then as a Manager of the Development Team. Prior to this, Mr. Zeleny has worked as a Sr. NT Systems Engineer, IT Supervisor, Network Administrator and Sr. Technical Support Analyst. Mr. Zeleny has been employed at Air Toxics Limited since August, 2005.

**JEFFREY TECSON**  
**SUPPORT SERVICES TEAM LEADER**

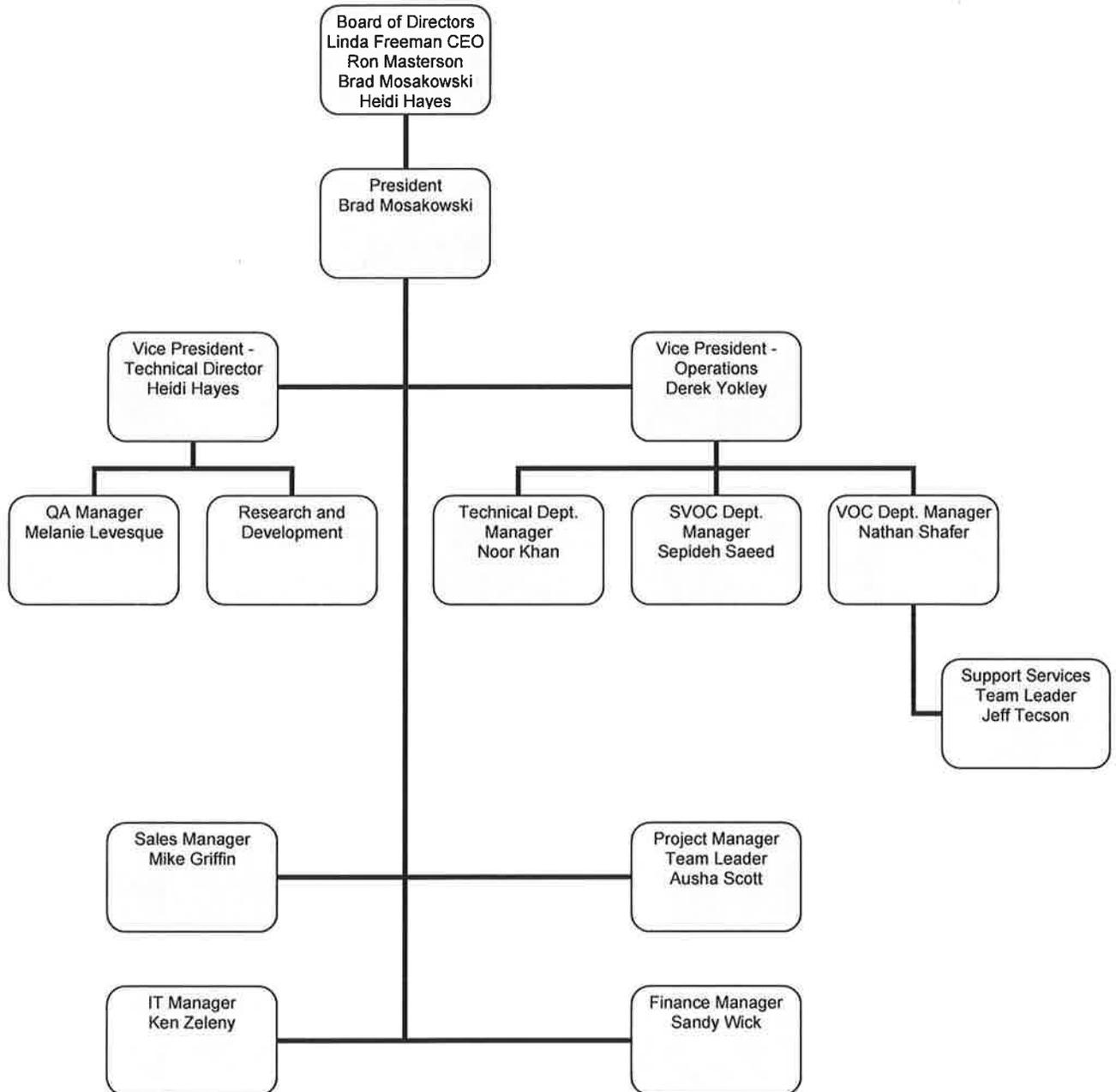
Mr. Jeffrey Tecson is the Team Leader for the Support Services Team. This team is

responsible for cleaning and coordinating the certification of Summa, Silco and Silonite Canisters. Other responsibilities include preparation of flow controllers, TO-17 tubes, VOST/SMVOC tubes for Methods 0030 and 0031. Mr. Tecson has 7 years experience in doing bench work for Support Services; currently Mr. Tecson is spending 25% of his time on the bench. Mr. Tecson has an A.S. Degree in Computer Technology Heald College in Rancho Cordova, CA; he also has 13 years management experience.

**AUSHA SCOTT**  
**PROJECT MANAGER**  
**TEAM LEADER**

Ms. Ausha Scott is the Team Leader for the Project Management Department. She is responsible for overseeing the project management functions, including client relations and technical support. Ms. Scott has 7 years of environmental laboratory experience in a variety of positions including GC/MS chemist and client service representative. Ms. Scott holds a Bachelor's degree in Marine Biology from University of California, Santa Cruz.

**Exhibit 2.1. ATL Management Organization**



## **2.2 FACILITIES**

The ATL laboratory occupies 30,000 square feet of space in Folsom, California with approximately 6,000 square feet of office space. The single story building is custom designed to suit the specifications of an air laboratory. Design criteria included floor plans to accommodate segregation of conflicting tests and provide an environment that is conducive for cross-functional work teams. The main instrumentation laboratory is based on an "open" concept in which walls are removed to promote a sense of community and teamwork. Wide hallways with alcoves are designed to encourage congregation and discussion. The number of private offices is minimized so that barriers between management and staff are removed. Elements of the quality system are evident throughout the facility design.

Sample receiving occupies approximately 950 square feet. There is sufficient floor space to receive, unpack, and tag up to 150 Summa™ canisters per day. The main laboratory is centrally located and houses twenty one GC/MS systems, eleven GCs, and a network of computers.

A caged canister storage area was constructed on one side of the laboratory to securely hold all canister and Tedlar bag samples. An isolated negative pressure room was designed for solvent handling and extraction activities. Approximately 1000 square feet of air-conditioned space is designated for research and development activities, and a work shop/tooling area. Sorbent tube preparation and canister cleaning operations are located in segregated areas. Long-term file storage occurs off site. A local document storage and retrieval service picks up files for storage.

Files are kept in bar coded boxes making retrieval easier. Typically a file can be retrieved within one working day from the original request.

Security is maintained through a controlled access system. Representatives of State, Federal or private entities have access to the laboratory facility and records during laboratory normal business hours. Guests must enter/exit through a central reception area. The receptionist keeps a date/time log. After work hours, the building is secured and linked to a commercial security agency. The security system is equipped with perimeter alarms, motion sensors, and speakers that monitor background sounds. Heat activated fire alarms are monitored by an outside agency. A fire alarm also activates the security system. ATL SOP #30 describes the security and controlled access protocols.

## **2.3 EQUIPMENT AND INSTRUMENTATION**

The laboratory is equipped with over \$2,000,000 of instrumentation, dedicated exclusively to the analysis of air samples. Much of the commercially available equipment is modified in-house in order to enhance performance in the areas of:

- *Overcoming challenging sampling problems;*
- *Analyzing difficult matrices;*
- *Achieving greater sensitivity.*

A staff of design engineers and a 1,000 square feet fabrication shop is maintained by ATL in order to build, test, and service the custom equipment. A facilities map and equipment list can be found in Exhibit 2.2 and Tables 2.1 and 2.2.

Exhibit 2.2. Facilities Map

Air Toxics 180 Blue Ravine Rd. Suite B

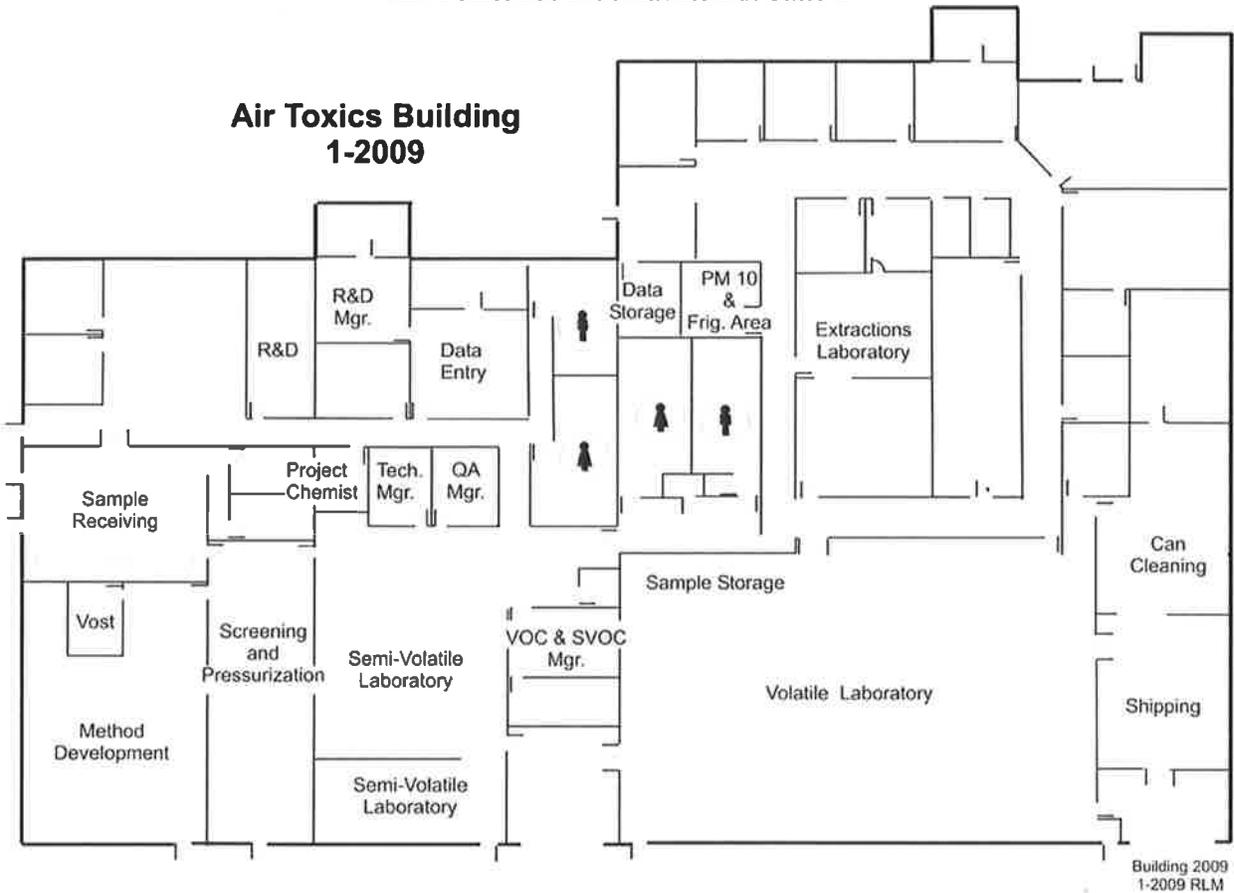
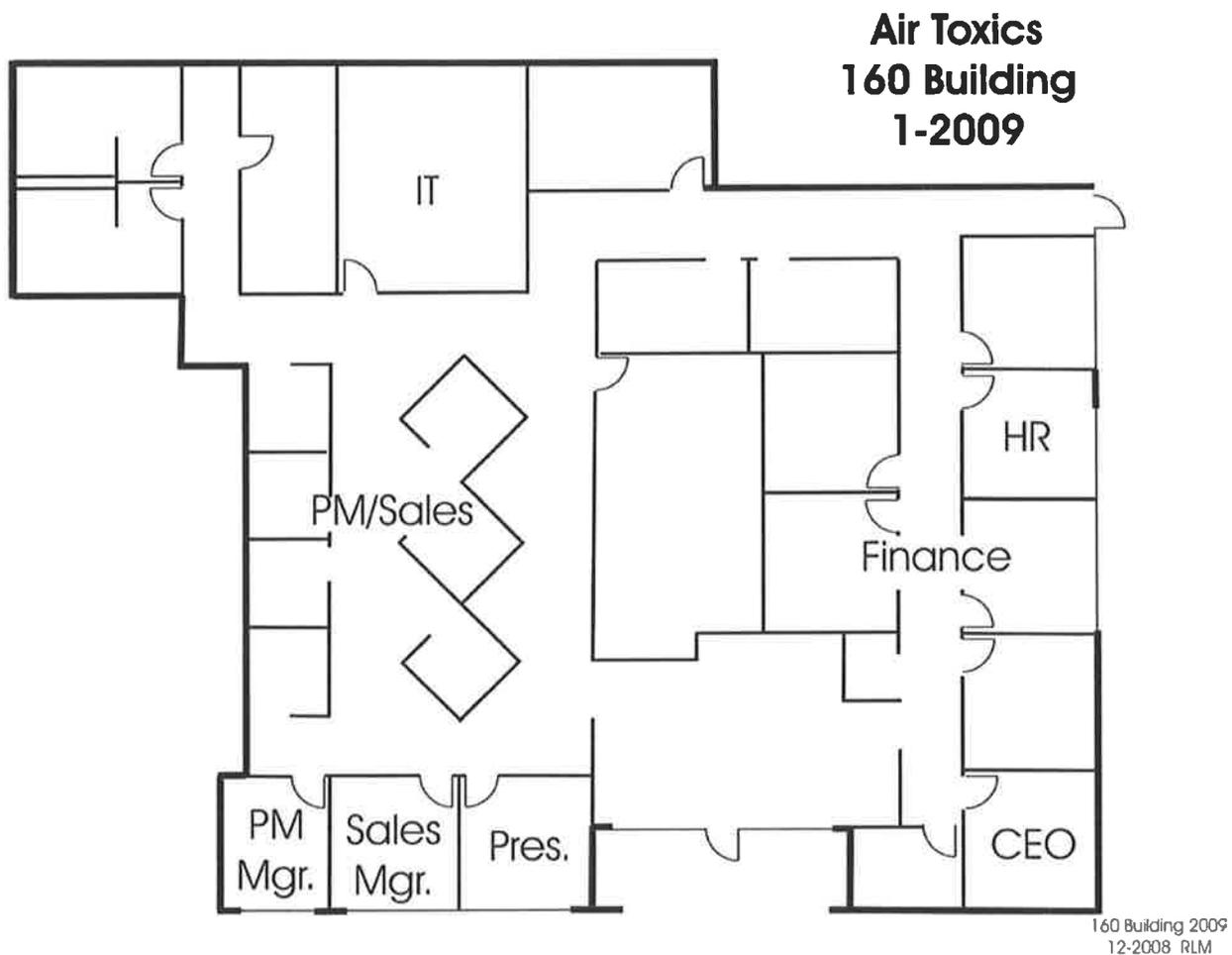


Exhibit 2.2. Facilities Map (Continued)

Air Toxics 160 Blue Ravine Rd. Suite A



**Table 2.1. Laboratory Instrumentation and Equipment**

#	Description
1	Agilent 5972 GC/MSD
6	Agilent 5973 GC/MSD
13	Agilent 5975 GC/MSD
1	Leco Time of Flight MS
1	Markes Autosampler
1	Hewlett-Packard 5980 GC/ECD/ECD
2	Hewlett-Packard 5980 GC/SCD
3	Hewlett-Packard 5890 GC/FID
2	Hewlett-Packard 5890 GC/TCD/FID
2	Hewlett-Packard 6890 GC/PID/FID
1	Hewlett-Packard 5890 GC/TCD/ECD
1	Hewlett-Packard 1200 Gradient HPLC
6	Air Toxics Canister Autosampler
2	Air Toxics Custom Sorbent Tube Desorption Unit
8	Canister pressurization stations
15	Soxhlets Extractors
1	Automated Canister CART Cleaning Station
2	Accelerated Solvent Extraction (ASE) System
4	Custom Pressure/Vacuum Canister Cleaning Manifold
10	Custom Convector Vacuum Canister Cleaning Manifold

**Table 2.2. Sampling Media**

Description	Quantity
Air sampling canisters	
6-Liter Summa canister	2350
1-Liter Summa canister	1500
PAC250 Summa Canister	440
High Pressure Sample Cylinder	30
Flow Controllers for air sampling canisters	800
24-hour flow controllers for canisters	750
Vacuum gauges	200
Tedlar bags: 1, 3, 10 liter	In inventory
MM5 air sampling traps	10
Midget impingers	30
VOST tubes kept in inventory	50 pair
TO-13 PUF/XAD and TO-4/TO-10 air sampling cartridges	200
TO-17 air sampling tubes various sorbents	450

### 3.0 QUALITY PROGRAM PLAN

Air Toxics Limited maintains comprehensive Quality Assurance programs to ensure that analyses are being conducted according to prescribed analytical methodology, and are within project specific QAPP requirements. The program is an integrated system of activities involving planning, quality assessment, quality control, reporting, and quality improvement. The basic elements of this program include:

- **DOCUMENTING** procedures, method requirements, and project requirements
- Organizing, monitoring, and leading **TRAINING** programs on quality related issues
- **ASSESSING** adherence to requirements, including maintenance of a system which documents, tracks, and provides closure when corrective actions are necessary
- Formally **COMMUNICATING** results of those assessments to laboratory management

These critical elements of the Quality Plan are described in detail in the following sections.

#### 3.1 DOCUMENTING

##### 3.1.1 The Quality Assurance Manual

The Quality Assurance Manual describes the major programs or systems by which the laboratory provides data of known and predictable quality. The QA Manager and the Laboratory Director are responsible for the content, accuracy and completeness of the Manual. The Manual must comply with all State and Federal requirements for those programs in which the laboratory maintains accreditation. The Quality Assurance Manual is a required reading for all laboratory staff and everyone must comply with the

procedures documented as a condition of continued employment. The QA personnel inform the Department Managers and the Team Leaders of the availability of the revised Quality Assurance Manual. The Department Managers or the Team Leaders inform team members who then access the Manual from the network in order to read the Manual. Each staff person documents in his/her training record that the latest revision Manual has been read and understood. The Department Manager and/or the Team Leader assess the accuracy and completeness of the documentation annually at the time of the employee's performance review. Missing or incomplete documentation is noted in the performance review.

The Quality Assurance Manual is reviewed and updated annually. The Manual is signed and dated by the Technical Directors, the Vice President of Operations and the Quality Assurance Manager as acknowledgment of review and approval prior to use. All personnel are required to document reading the latest version in their training record.

##### 3.1.2 Standard Operating Procedures/Methods Manual

The laboratory procedures used at ATL are documented in method-specific standard operating procedures (SOPs). These procedures are based on standard EPA or ASTM methodology whenever possible. The SOPs contain all necessary QC parameters, acceptance criteria, and directions for corrective action measures. The SOPs govern the laboratory response to results that are outside acceptance limits and address anticipated problems with associated recommended corrective action to eliminate the problem or further occurrences of the problem. SOPs also specify the type of written records (typically Corrective Action Requests known as CARs) necessary to fully document anticipated as well as unanticipated problems. The SOPs are maintained in numerical order in binders, which also serve as

the laboratories Methods Manual. The SOPs address the following (where applicable):

- Identification of the test method
- Applicable matrix or matrices
- Limit of Detection (MDL)
- Method reporting limits
- Scope and application (includes target analytes)
- Summary of the method
- Table of significant variances from the method
- Definitions and interferences
- Safety
- Equipment and supplies
- Reagents and calibration standards
- Sample preservation and storage requirements
- Quality control
- Calibration, validation, and standardization
- Procedures
- Data analysis and calculations
- Method performance objectives
- Pollution prevention (if applicable)
- Data review and acceptance QC criteria
- Corrective Action for out of control data
- Waste management (if applicable)
- Method identifier and references
- Any relevant tables, flow charts or diagrams

The SOPs are written by the Department Manager, the Team Leader or an experienced Scientist/Analyst and are reviewed annually for technical accuracy and adherence to general QA/QC protocols. The SOP is signed and dated by the author, then is submitted for technical review, QA review, and final review by the Laboratory Director. Each method SOP contains a detailed table of all modifications taken against the actual reference method. Modifications and/or additions to the SOP are similarly reviewed and signed. Each hard copy SOP carries a unique revision number, control copy number, and date of generation. The SOPs are treated

as confidential and proprietary and are maintained under the authority of the QA Department. The original is kept in the QA Department and extra copies in various laboratory sections as needed. Electronic versions of the SOPs are stored on a secured network drive, which only the QA Department can access. SOP summaries that include analyte lists, reporting limits, QC criteria and current variances to published methods are available to clients in .pdf form as the ATL Methods Manual Summary. Copies of SOPs are made available to State and Federal accreditation and regulatory entities.

Current SOPs are stored electronically in a secured read-only database to allow review online by laboratory personnel. Whenever an SOP is updated and implemented, it appears in the electronic database. The QA personnel inform the Department Managers and the Team Leaders of the availability of the revised SOP. The Department Managers or the Team Leaders inform team members who then access the SOP from the laboratory network in order to read the SOP. Once the Scientist/Analyst reads the SOP, he/she logs the date in the SOP tracking software, or signs and dates a copy of the title page from the hard copy stored in the laboratory. This documentation is then filed in his/her training record. The Department Manager and/or the Team Leader assess the accuracy and completeness of the documentation annually at the time of the employee's performance review. Missing or incomplete documentation is noted in the employee's performance review.

A comprehensive list of ATL's SOPs can be found in Appendix B.

### **3.1.3 Revisions to SOPs**

The revision number of the referenced method is noted in the method-specific SOPs. The protocols and deviations are specific to that revision number. Air Toxics does not operate under more than one version of a referenced

method at any time. The specific protocol used for analysis can be tracked using the effective date noted on the front page of the SOP.

Each SOP update is identified by a unique revision number. As with referenced method revisions, only one revision of an SOP is used in the laboratory at any one time. A complete description of ATL's system for writing and updating SOPs can be found in ATL's SOP #46.

**3.1.4 Documenting Method Specific Deviations**

Most air methods were not written as definitive and all have a strong performance based component to them. It is not unusual for the lab to have to design and create sampling interfaces or moisture control devices or to add additional quality assurance requirements to the methods in order to meet more stringent project or program requirements. Any variances to referenced methods are summarized in tabular form in the laboratory SOP. Signatures of the Laboratory Director and QA Manager on the front page of each SOP indicate review and approval of

these variances. A copy of the method modifications table from the SOP appears in the Laboratory Narrative section of the Comprehensive Validation Package or standard final report (Table 3.1). The QA team maintains and updates the templates used to create the Laboratory Narrative section of each work order. Each template has a revision date to ensure that only the most recent SOP table appears in the Laboratory Narrative.

On occasion, the need arises to change some aspect of an established SOP to accommodate enhancements to either the equipment or the method. The bench chemist documents the request for the change and the reasons behind it in the 'Request for Technical Change' form (Exhibit 3.1). The form is routed through the Department Manager and Team Leader with approval for the change noted by signature of the Laboratory Director and Technical Director. The form also identifies any established SOPs which should be revised/amended to incorporate the changes made. The forms are serialized in order to track progress and implementation. SOP Amendment forms are also used to reflect changes that are made to SOPs prior to a new revision.

**Table 3.1. Example Method Modification Table**

<b>Requirement</b>	<b>EPA Method TO-4A/TO-10A</b>	<b>ATL Modifications</b>
Extraction Solvent	10 % (5 % TO-10A) Diethyl Ether in Hexane	DCM, exchanging to Hexane during the concentration step
Reagent Blank	Set up extraction system without filter/PUF; reflux with solvent	No Reagent Blank is extracted. Reagent lots are certified as acceptable prior to use
Media certification (TO-10A only)	< 0.01 ug for single peak analytes, < 0.1 ug for PCBs	< Reporting Limit for all analytes
Frequency of Continuing Calibration Verification	Every 10 samples	Every 20 samples with internal standard
PCB Quantitation	Requires a minimum of 5 peaks	Use 4 peaks for quantitation

**Exhibit 3.1. Example Technical Change Request Form**

**Request for Technical Change Form**

No. VOC -001

*This form is to be used to propose any technical changes from ATL's Standard Operating Procedures.*

Initiated By: \_\_\_\_\_ Date: \_\_\_\_\_

**I. Description of Proposed Change:**

---

---

---

---

---

---

---

---

**II. Reasons for Proposed Change:**

---

---

---

---

---

---

---

---

**III. Method/Instruments Affected:**

---

---

**Exhibit 3.1. Example Technical Change Request Form (Continued)**

**IV. Approvals:**

Laboratory Director Approval: \_\_\_\_\_  
 Linda L. Freeman \_\_\_\_\_ Date \_\_\_\_\_

Technical Director Approval: \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

**V. Changes in ATLAS Database**

Valid Values Table(s)/Analyte Lists changed by: \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

**VI. Affected SOPs:**

SOP/Rev #: \_\_\_\_\_ Date Revised/Amended: \_\_\_\_\_ Revised/Amended by: \_\_\_\_\_  
 Initials \_\_\_\_\_

SOP/Rev #: \_\_\_\_\_ Date Revised/Amended: \_\_\_\_\_ Revised/Amended by: \_\_\_\_\_  
 Initials \_\_\_\_\_

SOP/Rev #: \_\_\_\_\_ Date Revised/Amended: \_\_\_\_\_ Revised/Amended by: \_\_\_\_\_  
 Initials \_\_\_\_\_

**VII. Additional Comments:**

\_\_\_\_\_  
 \_\_\_\_\_

Initials: \_\_\_\_\_ Date: \_\_\_\_\_

**VIII. Notifications:**

QA Manager: \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

Department Manager: \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

CSR Team Leader (as required): \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

Team Leader (as required): \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

Team Leader (as required): \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

Team Leader (as required): \_\_\_\_\_  
 Signature \_\_\_\_\_ Date \_\_\_\_\_

**Date Implemented:**

This change to ATL's Standard Operating Procedures has been implemented by:

\_\_\_\_\_  
 QA Signature \_\_\_\_\_ Date \_\_\_\_\_

**3.1.5 Documenting Project Specific Deviations**

Project specific QAPPs are reviewed by the QA Department including the Project Chemist (or a qualified designee). The laboratory may also take variances against method criteria established in project specific SOWs or QAPPs. The Project Chemist (or qualified designee) reviews the project specific criteria during project proposal and notes any variances from standard laboratory SOP in a table. A variance table (Table 3.2) is then incorporated into the bid proposal for review and acceptance by the prospective client. The client notes acceptance by signature or initial and date in the designated field of the table.

The project specific variance table is stored on a secured network drive following approval. A Project Profile (Exhibit 3.2) is initiated at the same time. A summary of the analytical requirements which differ from ATL's relevant SOP is documented in a Project Requirement Table and included in each Work Order folder. The Project Profile may also be accessed through the ATLAS database. The Project Manager is responsible for noting the location of the Project Requirement Table in the Project Profile. Finally, the variance table is included in the Comprehensive Validation Package (CVP). The project specific QAPPs are maintained in electronic form on a network drive.

**Table 3.2. Example Project Specific Variance Table**

SOW	ATL SOP	VARIANCE APPROVAL*
Method 2720C Fixed Gases	ASTM D-1945 Fixed Gases	
RDLs determined upon receipt of lab MDLs	Standard lab RDLs of 10 ppmv	
Field blank can one per batch	Not provided	
Canister released 90 days past reporting	Canisters released 30 days past day of sampling	
72 hour retention time study	±0.06 minutes standard SW-846	
Calibration verification daily with all analytes ±25% expected value	All compounds within 15%; Spike concentration is 25 ppbv	
Accuracy/precision study per analyst with project limits	Per analyst once every 12 months using ATL standard limits only	
LCS once per 5 point, ±25% difference for all compounds	All compounds within ±15%; Spike concentration is 25 ppbv	

\*NOTE: Each variance needs to be approved by the client's initial and date. An initialed copy of this variance table will appear in the Comprehensive Validation Package.

## 3.2 TRAINING

### 3.2.1 Team Training

ATL laboratory staff members have sufficient education, training, and technical knowledge to perform their assigned duties. Each team has both experienced and in-training staff members. Those in training work under the supervision of a more experienced peer who is typically the lead Scientist assigned to that area.

Training of laboratory staff in analysis consists of three developmental stages:

- STAGE I. Introduction

Initial instruction by the Department Manager or a designated peer concerning basic elements of the method and brief overview of instrumentation. Applicable SOPs and methods are read. During this time, the trainee is an observer.

- STAGE II. Training

Periods of close contact and direct supervision by the Department Manager or a designated peer. During this time, which may last for several weeks, the Analyst/Scientist performs tasks independently as assigned. All aspects of his/her work are reviewed by the Department Manager or a designated peer.

- STAGE III. Advanced Operation

Independent work with data review by the analytical Department Manager or a designated peer.

The final step in the training process allows the Analyst/Scientist to document competency by analyzing four consecutive Laboratory Control Samples which is documented by a Demonstration of Capability Form for Initial Method Proficiency (see Exhibit 3.5). A Continuing Demonstration of Capability must be made on an annual basis and documented by a Continued Method Proficiency Form (see Exhibit 3.6). Personnel who perform on odd shifts and do not commonly spec out

instrumentation may substitute a duplicate analysis with acceptable precision (%RPD) for the four Laboratory Control Sample analyses when fulfilling the Continuing Demonstration of Capability.

The QA team ensures that training of each member of the technical staff is complete, documented, and up to date. Exhibit 3.4 is an example of a Training Record form. It is the responsibility of the employee to keep his/her record current. An Analyst's/Scientist's training is considered current if the training record contains evidence that the employee has:

#### Training Record Checklist

- QA Orientation documentation
- Safety Orientation documentation
- Read the current version of the LQAP (QA Manual)
- Completed the training class on ethical responsibilities
- Read and understood the current version of relevant SOPs
- Completed necessary internal or external training classes
- demonstrated initial proficiency in the methods by acceptable performance on four Laboratory Control Samples; proficiency is measured by accuracy and precision
- demonstrated continued proficiency in the methods by acceptable performance on four Laboratory Control Samples or duplicate analysis with  $RPD \leq 25\%$  between two analysts; proficiency is measured by accuracy and precision

The Department Manager and/or Team Leader review the training record on a yearly basis during an employee's annual performance review. Deficiencies in the training record are documented and returned for correction (see Exhibit 3.7). Consistent failure to maintain

updated training records is noted in the performance review and effects the employees overall job rating.

A series of classes taught by members of the management team are typically offered on a yearly basis. Exhibit 3.8 contains a sample of internal courses that are offered. The list of courses is subject to change on a yearly basis depending on requests and topics. Course attendance is mandatory for topics specifically related to an employee's job function. The QA Department and management teams determine which courses are mandatory. Course attendees may be tested to ensure that they have achieved an acceptable understanding of the material presented. Completion of courses is documented in the employee's training record.

### **3.2.2 External Training**

External training courses offered by software experts, instrument manufacturers, or other recognized experts in analytical instrumentation and/or analysis are attended by ATL employees. The course description, dates offered and record of attendance are kept in the employee's training records. The company maintains a budget for external training classes and higher education.

### **3.2.3 Quality Training**

All new Air Toxics Limited employees are required to attend the Quality Assurance Orientation course. Completion of the course is documented in the employee's training record. The course outline includes:

- Introduction to QA and Laboratory Nomenclature
- Definitions of SOPs and LQAP
- How to use CARs
- Logbook protocol
- Chain of Custody procedures
- Training documentation
- ATL classes

- Ethics and Integrity I (Overview)
- Ethics and Integrity II (Annual )
- Overall Training Record organization and upkeep

### **3.2.4 Health and Safety Training**

Laboratory staff may, on occasion, be exposed to the handling of flammable solvents, compressed gases or toxic calibration standards. There are four to six staff members comprising the Safety Committee. Some members are 40 hour OSHA trained and respirator fitted. Education in the safe handling and disposal of these materials is accomplished as follows:

- Each new employee is given a safety tour of the facility within the first two weeks of employment. Documentation of this orientation appears in the employee's training record.
- The Safety Committee meets quarterly (or more frequently if needed) to discuss safety concerns and ways of improving safety in the work place.
- The Safety Committee schedules on going safety training throughout the year.
- If special precautions must be taken to perform a method, a safety section is included in the method SOP which discusses protocols and other measures for risk reduction through exposure prevention.
- ATL maintains Material Safety Data Sheets (MSDS) for each chemical used on-site. The MSDS are accessible to all personnel in the library area.
- ATL has access to MSDSs on the Internet through its vendors.

The Safety Committee staff members are assigned to duties including hazardous waste

disposal, incident or spill management, scheduling staff training, safety site assessments, Chemical Hygiene Plan review, and the overall leadership of the safety program.

### **3.3 ASSESSING ADHERENCE AND COMMUNICATING FINDINGS**

The QA team plays a key role in establishing quality policy and protocols. The QA Department ensures that the established guidelines are followed through various quality control programs, which are designed to detect non-compliance or departure from protocol. Each quality control program includes documentation of the assessment process and timely feedback to the management and staff involved.

#### **3.3.1 Data Review**

The QA team reviews Work Orders which the client has requested 100% QA review. For work that falls under the scope of the DoD, the QA team reviews 10% of the Work Orders. Deficiencies noted during review are documented and communicated to the staff involved.

#### **QA REVIEW CHECKLIST**

- Assessing Analysis/Reporting vs. Project Profile/SOP requirements**
- Verification of reporting list, units and report header information**
- Assessing accuracy and completeness of the laboratory narrative**
- Documentation of any corrective actions**
- Documentation of unusual circumstances**
- Verification of the QC meeting criteria**
- Verification of sample holding time**
- Verification of appropriate data flags**
- Appropriate peak integration and documentation for manual integration**
- Verification of adherence to analytical sequence clock times**
- Verification of the appropriate Initial Calibration**
- Manual verification of one sample result from raw area counts**

- Verification of sample dilution factors**
- Checked samples for trends**
- Verification of sample id's vs. COC**
- Assessing accuracy and completeness of the Client Lumen report (if applicable)**

The QA reviewer will look both for appropriate as well as inappropriate laboratory practices. Inappropriate practices are those which fall outside established laboratory SOPs. If inappropriate practices are suspected, the QA reviewer will verify the result with the QA Manager and the Department Manager, initiate a Corrective Action Request, and if necessary, form a committee consisting of but not limited to the Department Manager, Technical Director, and Laboratory Director. Most Corrective Action Requests may be traced to human error. Oversights of this nature are simply documented and feedback is given to the Analyst or Scientist involved.

On occasion, the committee may determine that the Corrective Action Request was not attributable to simple human error. The reasons for the non-conformance and appropriate action to be taken are discussed and implemented by this committee. Typical actions include retraining of the individual involved along with a remedial period of close monitoring by the Department Manager. The QA team or an approved peer reviews all data reported by the Analyst during the remedial period. The laboratory uses a three-strike rule with respect to non-compliance issues. New Analysts are rigorously trained to follow SOPs. This training lasts several months. Retraining is done if there is cause to suspect non-compliance. Secondary training typically lasts 30 days. Any further evidence of non-compliance may result in termination.

Project Managers create Project Profiles that specify 100% QA review. When samples are received the Sample Receiving Team will automatically add a QA review to a Work Order if 100% frequency has been requested as per the profile.

After QA review is complete the reviewer enters the review date in the sample tracking database. If a QA reviewer discovers an error, necessary corrections are made and the work order is reissued.

Another tool used for data review involves the use of proprietary in-house data validation software to review every data point generated and to alert the reviewer when manual integrations occur. The software is also programmed to report when more than three attempts for a daily CCV or tune check standard to pass have been attempted. (Validation software currently reviews all method TO-14A/TO-15, TO-17, ASTM D-5504, TO-3, TO-12 and SW8260B results. Further software development is currently ongoing to bring more methods on line).

### **3.3.2 Corrective Action Program**

The QA Department manages the Corrective Action Program and maintains the Corrective Action tracking database using software called c.Support. A Corrective Action Request (CAR) is initiated any time sample results are adversely affected by system non-compliance with established SOPs or program requirements, any time an internal or external assessment results in a finding, any time there is a failed proficiency evaluation sample, any time there is a failure quality system such that data quality is affected, and lastly, any time there is a customer inquiry into the laboratory's data quality and laboratory error is found (see Exhibit 3.2). This request is documented in the CAR database using the c.Support software (see Exhibit 3.9). The database is also used to track the date of resolution, the necessity for a follow-up, and the date the follow-up action is completed.

Corrective Action Requests which require immediate resolution must be completed and finalized within 2 business days. All other CARs must be resolved within 30 business days. The status of corrective actions that have not yet been completely resolved

(including follow-up actions), are discussed during the bi-weekly QA meetings. Whenever a customer raises an issue relating to data quality, the inquiry is documented in the Client Contacts database. A representative of the QA team or a Technical Director reviews the data in question and investigates any systematic problem that may be evident. If results of the review and investigation merit corrective action a CAR will be initiated along with any necessary follow-up action.

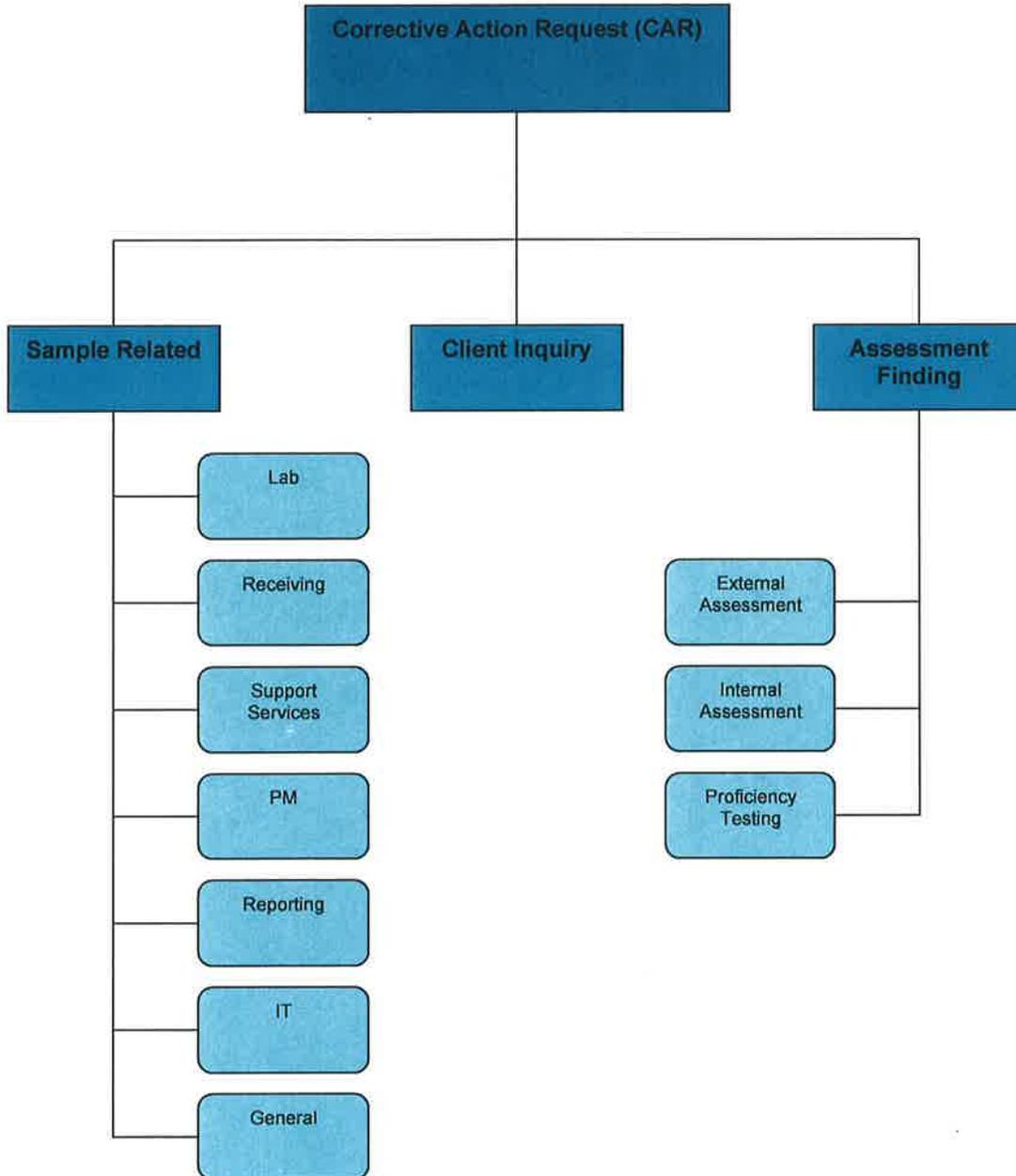
Examples of when the client inquiry CAR may be initiated include:

- Blind field duplicates that do not agree
- Field blanks that had contamination present.
- Blind proficiency sample that did not meet accuracy objectives
- Sample splits that do not meet precision objectives
- Outlet sample results that were higher than inlet sample results
- Sample results that cannot be manually verified
- Sample results that do not meet program requirements

A portion of the CAR database is associated with sample receiving and analysis. Should a malfunction occur with a pending sample, the client is contacted prior to analysis to confirm if the analysis should continue. The CAR documents the contact and resolution of the issue. Should the decision be made to proceed with analysis then any malfunction affecting data quality is detailed in the laboratory narrative. Instructions to proceed with analysis and narration of the affected results are documented in the CAR. The Department Manager and/or Team Leader must review the CAR and determine if the error is isolated or systematic. If the CAR casts doubts on the laboratory's compliance with its quality assurance program or the NELAP and DoD QSM standards, the QA Manager will audit the lab teams practices as soon as possible. After QA review, the CAR is then filed with

the Work Order as a permanent record of the nature of the problem and the resolution. A complete description of ATL's CAR system can be found in ATL's SOP #61.

**Exhibit 3.2. Types of Corrective Action Requests**



### **3.3.3 QA Management Meetings**

Every two weeks the QA Manager leads a meeting with the Department Managers, the IT Manager, the Project Manager Team Leader, and the Support Services Team Leader. All other Managers and Directors are welcome to attend. These meetings are called to discuss the effectiveness of the quality systems, specific quality issues that may have surfaced from the time of the last meeting, and to monitor progress with respect to open Corrective Action items. Agenda items are added and removed at the discretion of the QA Manager or QA staff.

The meeting is used as an interactive forum in which non-compliance issues are discussed with respect to the overall suitability of the quality system involved. Non-compliances are screened to see if the quality system itself is in need of a review or modification. If it is determined that a particular quality system needs to be designed or revised then the committee takes responsibility for restructuring that system. The issue cannot be removed from the agenda until the new system is in place. Minutes of each meeting are kept in a QA electronic form on the QA network drive.

### **3.3.4 Conducting Internal Assessments**

The QA team conducts internal assessments of all major production areas of the lab on a yearly basis. The production areas are separated into assessment modules by referenced methodology. Whenever possible audits are scheduled to occur after the yearly update and revision of the relevant SOP. Audits are composed of three events:

- Laboratory assessment based on the current SOP by the QA Team.
- Circulation of the assessment report and issuing of any necessary corrective action forms.

- Satisfactory response to audit findings and verification of the implementation and effectiveness of the corrective action taken.

An assessment checklist is developed for each area by the QA Manager or designated staff. The checklist contains general, method specific, and SOP specific practices (Exhibit 3.10). The assessment process addresses whether or not quality systems (e.g., adherence to the current revision of the SOP, proper and complete documentation practices etc.) are in place and understood. Health and safety issues are also covered.

Results of the assessment are summarized in the checklist which also serves as the report. Findings that are determined to be in need of Corrective Action are processed through the standard CAR program. If findings imply that there has been a significant impact on the data, the report will be corrected and reissued to the client. Copies of the internal assessment report are circulated to the Department Managers and Team Leaders and other members of ATL management team as needed.

### **3.4 COMMUNICATING WITH MANAGEMENT**

The Quarterly QA Status Report summarizes the results of internal and external assessments, the numbers and types of CARs produced, the status of any outstanding CARs, a summary of customer inquiries received, PT results, and the number and types of reissued sample reports. This report is distributed to all Directors, Managers, and Team Leaders.

Exhibit 3.3. Example ATL Project Profile

**Project Profile**

Project Name

Big Landfill

Project Number

P.O.#

Project Description

Project Requirement Table: O:\Variances\2003\0303-001QC

Report to Address

Name: Mr. John Jones  
 Company: Average Engineering Firm  
 Address: 1234 Anystreet Avenue

Bill to Address

Mr. John Jones  
 Average Engineering Firm  
 1234 Anystreet Avenue  
 Your Town IA 50841

City:	Your Town	Done:	Y	QAPP on File?	Y
State/Zip:	IA 50841	CS Rep:	DD	Penalties?	N
Phone/Fax:	641-987-6543 (Phone) 641-234-5678 (Fax)	ProjectID:	5273	24 Hour Clock?	Y
Email:	jj@aef.com	Variance:	Yes	Charge For Shipping?	N

Analysis(es):	Reporting List:	Units	TAT	Price	Surcharge
Modified TO-15	Modified TO-15	ppbv	10 Day	.00	None

QA/QC:

Reporting List	Dups	CCV	LCS
Modified TO-15	10%	10%	1/ANB

Media:	Media Price:
6 Liter Summa Canister	.00

Deliverables:	Done:	Price:	Price Is:	Due:	Send VIA:
Standard ATL Report	<input type="checkbox"/>	.00	Per WO	10 Working Days	
Client Specific Disk Format	<input type="checkbox"/>	.00	Per WO		Mail

Notes to Receiving:

Notes to Lab:

Notes to Reporting:

Miscellaneous Items:

Price:

**Exhibit 3.4. Example Laboratory Training Record**

@ Air Toxics Ltd.

**LABORATORY TRAINING RECORD**

EMPLOYEE: \_\_\_\_\_ PROCEDURE: \_\_\_\_\_

The normal training program consists of three developmental stages, as explained below. Indicate the necessary dates completed for each stage. You should always progress from one stage to the next in sequential order. Training is complete when this form, along with associated precision data, is submitted to the Quality Assurance Department. Upon return this form should be retained in the employee training record.

**STAGE I. Introduction**

Initial instruction covers the basic elements of the method as well as a brief overview of the instrumentation and is provided by the Team Leader or an individual who has been previously qualified for training on the procedure. Activities include reading applicable SOP(s), reading the reference method, and loading samples. During this time, the trainee is an observer.

Date Initiated: \_\_\_\_\_ Trainer: \_\_\_\_\_  
Date Completed: \_\_\_\_\_ Trainers Qualification Date: \_\_\_\_\_

**STAGE II. Training**

This is a period of close contact with and one-on-one training by the Team Leader or a qualified peer. Activities include performing Initial and Continuing Calibrations, routine maintenance, and data reporting. During this time the trainee performs tasks independently as assigned but with all aspects of his/her work reviewed by the Team Leader or qualified peer.

Date Initiated: \_\_\_\_\_ Trainer: \_\_\_\_\_  
Date Completed: \_\_\_\_\_ Trainers Qualification Date: \_\_\_\_\_

**Stage III. Advanced Operation**

During this period the trainee operates completely independently with his/her work passed into the normal review cycle. Completion of this stage is accomplished when four LCS standards are analyzed and meet the method accuracy and precision objectives (attach quant sheet(s) and/or summary and copy of run logbook/extraction logbook page(s)).

Date Initiated: \_\_\_\_\_  
Date Completed: \_\_\_\_\_

Employee Signature: \_\_\_\_\_

**Training Approval**

The three stages of training have been completed and the employee considered to be qualified to perform the procedure.

TEAM LEADER: \_\_\_\_\_ Date: \_\_\_\_\_  
QUALITY ASSURANCE: \_\_\_\_\_ Date: \_\_\_\_\_



**Exhibit 3.6. Continued Method Proficiency Form**

@ Air Toxics Ltd.

**Demonstration of Capability**

**Continued Method Proficiency Certification Statement**

Date Certified: \_\_\_\_\_

Laboratory Name: Air Toxics Limited  
 Address: 180 Blue Ravine Road, # B  
Folsom, CA 95630

Matrix: Air  
 Method Name: \_\_\_\_\_  
 SOP: \_\_\_\_\_  
 File Numbers: \_\_\_\_\_

We, the undersigned, CERTIFY that:

Analyst/Scientist	Signature	Date
-------------------	-----------	------

1. The analyst identified above, using the cited test method(s), which is in use at this facility for the analyses of samples under the National Environmental Laboratory Accreditation Program, have met the Continued Demonstration of Capability.
2. The test method(s) was performed by the analyst identified on this certificate.
3. A copy of the test method(s) and the laboratory specific SOPs are available for all personnel on site.
4. The data associated with the Continued Demonstration of Capability are true, accurate, complete and self-explanatory (1).
5. 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 well-organized and available for review by authorized assessors.

<u>Heidi C. Hayes</u>		
VP/Technical Director	Signature	Date

<u>Melanie A. Levesque</u>		
Quality Assurance Manager	Signature	Date

This certification form must be completed each time a capability study is completed.

- |                   |  |
|-------------------|--|
| (1) True:         | consistent with supporting data  |
| Accurate:         | based on good laboratory practices consistent with sound scientific principles and practices.        |
| Complete:         | includes the results of all supporting performance testing   |
| Self-explanatory: | data properly labeled and stored so that the results are clear and require no additional explanation |

Revised 8/1/2007

**Exhibit 3.7. Example Training Record Review Check Sheet**

@ Air Toxics Ltd.

**TRAINING RECORD CONTENT SHEET**

Employee: \_\_\_\_\_ Review Date: \_\_\_\_\_  
Department: \_\_\_\_\_ Position: \_\_\_\_\_

**Required Documentation**

---

- Resume
- QA Orientation Training
- Safety Orientation checklist
- Documentation of Reading current version of the LQAP
- Current Ethics Training
- Documentation of reading current version of applicable SOP(s)/Amendments

**Documentation of Classes / Continuing Education Courses (completed since previous review)**

---

- Quality Assurance Class      Date: \_\_\_\_\_
- Manual Integration Protocol      Date: \_\_\_\_\_
- Hazard Communication Training      Date: \_\_\_\_\_
- Inappropriate Lab Practices      Date: \_\_\_\_\_
- Other: \_\_\_\_\_      Date: \_\_\_\_\_

**Other Documentation / Comments**

---

(notation of additional or missing items, incorrectly completed forms, organization, etc.)

- Documentation of Training attached for Methods/Procedures (please list)

\_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Review**

---

Employee Signature: \_\_\_\_\_ Date: \_\_\_\_\_  
Reviewed By: \_\_\_\_\_ Date: \_\_\_\_\_

**Exhibit 3.7. Example Training Record Review Check Sheet (Continued)**

@ Air Toxics Ltd.

**ADDITIONAL TRAINING RECORD CONTENT**

Employee: \_\_\_\_\_ Date: \_\_\_\_\_

Department: \_\_\_\_\_

**Documentation of Training (Continued)**

---

**Procedure:** \_\_\_\_\_

**Laboratory, Data Write-up, or General Training Record**

---

Completion Date: \_\_\_\_\_

- |  |  |
|--|--|
| <input type="checkbox"/> "Date Completed" and "Trainer" filled out for each stage<br><input type="checkbox"/> Employee Signature | <input type="checkbox"/> Team Leader Signature<br><input type="checkbox"/> Quality Assurance Signature |
|--|--|

**Initial Proficiency Data**

---

Completion Date: \_\_\_\_\_

- |  |   |
|--|---|
| <input type="checkbox"/> NELAP "Demonstration of Capability" form completed<br><input type="checkbox"/> Four replicate LCSs (analyzed on the same day or on separate days) | <input type="checkbox"/> Meets method criteria for Accuracy and Precision<br><input type="checkbox"/> Date and initials of reviewer on Proficiency Data |
|--|---|

**Current Continued Proficiency Data**

---

Completion Date: \_\_\_\_\_

- |   |  |
|---|--|
| <input type="checkbox"/> Current NELAP "Demonstration of Capability" form<br><input type="checkbox"/> Four replicate LCSs (analyzed on the same day or on separate days), or Duplicate Analysis (%RPD ≤ 25% between two Analysts) | <input type="checkbox"/> Meets method criteria for Accuracy and Precision<br><input type="checkbox"/> Date and initials of reviewer on Proficiency Data<br><input type="checkbox"/> Performed within a year of review date |
|---|--|

**Procedure:** \_\_\_\_\_

**Laboratory, Data Write-up, or General Training Record**

---

Completion Date: \_\_\_\_\_

- |  |  |
|--|--|
| <input type="checkbox"/> "Date Completed" and "Trainer" filled out for each stage<br><input type="checkbox"/> Employee Signature | <input type="checkbox"/> Team Leader Signature<br><input type="checkbox"/> Quality Assurance Signature |
|--|--|

**Initial Proficiency Data**

---

Completion Date: \_\_\_\_\_

- |  |   |
|--|---|
| <input type="checkbox"/> NELAP "Demonstration of Capability" form completed<br><input type="checkbox"/> Four replicate LCSs (analyzed on the same day or on separate days) | <input type="checkbox"/> Meets method criteria for Accuracy and Precision<br><input type="checkbox"/> Date and initials of reviewer on Proficiency Data |
|--|---|

**Current Continued Proficiency Data**

---

Completion Date: \_\_\_\_\_

- |   |  |
|---|--|
| <input type="checkbox"/> Current NELAP "Demonstration of Capability" form<br><input type="checkbox"/> Four replicate LCSs (analyzed on the same day or on separate days), or Duplicate Analysis (%RPD ≤ 25% between two Analysts) | <input type="checkbox"/> Meets method criteria for Accuracy and Precision<br><input type="checkbox"/> Date and initials of reviewer on Proficiency Data<br><input type="checkbox"/> Performed within a year of review date |
|---|--|

**Exhibit 3.8. Examples of Internal Training Courses**  
**(Subject to Change)**

<b>Quality Assurance</b>
Quality Assurance Orientation
Quality Assurance Protocols
Ethics and Integrity Training (I & II)
Inappropriate Laboratory Practices
Manual Integration Protocol
<b>Safety</b>
Health & Safety Training

### Exhibit 3.9. Example CAR

Problem - 947A2A4457

Page 1 of 1

Selection	New	Problem	Find
<div style="display: flex; justify-content: space-between; border-bottom: 1px solid black; padding-bottom: 5px;"> <span><b>Details</b></span> <span>History</span> <span>Assets</span> <span>Scans</span> <span>Custom Categories</span> <span>Others to Notify</span> <span>Attachments</span> <span>Associated Work Items</span> </div>			
Number: 947A2A4457		Impact: Lab	Status: Open
Assignee: Melanie Levesque		Urgency: 3 Day (3 Day Prop)	Publish to EUD: <input type="checkbox"/>
Opened: 4/7/2009 9:19:33 AM		Priority: High	
Categorization: CAR View Open Work Items for Categorization: Incidents Problems Changes Hide			
Short Description: CAR			
Description:			
Workaround:			
Root Cause:			
Resolution:			

**Exhibit 3.10. Example Internal Audit Checklist**

**Modified EPA Method TO-17  
 Technical & Quality Audit Checklist**

Date Audit Performed: \_\_\_\_\_  
 Auditor: \_\_\_\_\_  
 Participant(s): \_\_\_\_\_

Category	GLP/SOP Adherence Y/N/NA	Minor	Major	Comments
<b>Safety Procedures</b>				
Does the analyst have documented Safety Training?				
What is the proper procedure to follow when working with solvents or standards?				
<b>Sample Preservation, Handling and Storage</b>				
What are the holding times for TO-17 samples? At what temperature are the samples stored? How are these temperatures verified?				
How are tubes stored in order to prevent contamination?				
How is potential contamination monitored?				
How are samples logged into and out of the sample custody cage?				
<b>Interferences</b>				
What is the on-column concentration of a target analyte above which will trigger analysis of a system blank to eliminate possibility of carryover?				
What must be done when there is interference with the primary quantitation ion? What is the criterion for this to occur?				
<b>Instrument Operation</b>				
What are the different sorbent and which application are they used for?				
<b>Standard Preparation</b>				
How are these standards stored? What are the expiration dates of these standards?				
Provide documentation of standard preparation for TO-17 samples. Perform a manual re-calculation of a standard concentration. Trace a standard back to its certificate of analysis.				

**Exhibit 3.10. Example Internal Audit Checklist (Continued)**

**Modified EPA Method TO-17  
 Technical & Quality Audit Checklist**

<b>Calibration and Quality Control</b>			
How often must a BFB tune be analyzed? What if it fails acceptance criteria?			
At what level is the CCV analyzed? Is this level ever varied?			
When is an LCS analyzed and what are the acceptance criteria? Does the LCS contain all target analytes?			
What is the acceptance criterion for the Lab Blank? When is a Lab Blank analyzed?			
<b>Calculations</b>			
What is the equation for calculating a compound result? Show example.			
How do you calculate MDL values? Show example.			
<b>Sample Analysis</b>			
How does the analyst verify Project QC requirements prior to analysis?			
Verify the instrument operating parameters for TO-17 Methods. How are deviations from these parameters documented? Who is authorized to make these changes?			
What is the definition of an analytical batch?			
Review instrument Run Logbook. Has the logbook been reviewed on a monthly basis? Is documentation correct and complete?			
How are temperatures and flows used during analysis verified to be accurate?			
How are compounds qualitatively identified?			
How are compounds quantitatively identified? Re-calculate a sample result.			
Is it ever necessary to perform manual integrations? When is it acceptable or unacceptable to perform manual integrations? How must manual integrations be documented?			
<b>Data Review and Reporting</b>			
When is technical review necessary?			
Are flags applied to results in the final report verified at the time of management review?			

**Exhibit 3.10. Example Internal Audit Checklist (Continued)**

**Modified EPA Method TO-17  
 Technical & Quality Audit Checklist**

<b>Instrument Maintenance</b>				
How often does preventative maintenance occur? Who does this? Documented?				
How often is the vacuum gauge checked for potential system leaks?				
<b>General</b>				
Has it been documented in the analyst's Training Record that the current SOP for this procedure has been read and understood? (#5 Rev.7)				
Has the analyst demonstrated Proficiency for these methods within the last 12 months?				
Has it been documented that the analyst has read the current Laboratory QA Plan? (Rev. 19)				

Additional Comments: \_\_\_\_\_  
 \_\_\_\_\_

**A CAR has been generated for the Major findings. A response and completion of the CAR are due within 1 week of <enter date>.**

**A response for the Minor findings is due within 2 weeks of <enter date>.**

## **4.0 QUALITY OBJECTIVES**

The primary objective of the QA Program is to ensure that the laboratory is producing data that meet the laboratory's standard acceptance criteria for each method. Acceptance criteria from project-specific QAPPs are also used when required.

The laboratory's standard acceptance criteria and the sources of those criteria are specified throughout Section 6.0 of this Quality Manual. Definitions of parameters used to assess the quality of the data are defined below.

### **4.1 PRECISION, ACCURACY, REPRESENTATIVENESS, COMPLETENESS, AND COMPARABILITY**

#### **4.1.1 Precision**

Precision measures the reproducibility of measurements. Analytical precision is the agreement among duplicate (two) or replicate (more than two) analyses of the same sample. The acceptance for precision is determined using the relative percent difference (RPD) between the duplicate sample results. The %RSD (relative standard deviation) is used to document precision of linearity for the initial calibrations. The formula for the RPD and RSD calculations are provided in Exhibit 4.1.

Field duplicate samples represent *total* precision, the reproducibility associated with the entire sampling, and analysis process. However, the identification of field duplicate samples are typically not known to the laboratory, and therefore not specifically evaluated by the laboratory's QA department.

#### **4.1.2 Accuracy**

Accuracy measures correctness and includes components of random error (variability due to imprecision) and systemic error. Analytical accuracy is measured by comparing the

percent recovery of analytes spiked (as compared to the expected value) to pre-established accuracy limits (i.e., acceptance criteria). Any type of spiked sample measures accuracy. The formula for calculation of accuracy is included in Exhibit 4.1 as percent recovery (%R) from pure and sample matrices.

#### **4.1.3 Representativeness**

Representativeness is achieved through use of the standard analytical procedures described in this Quality Manual.

#### **4.1.4 Completeness**

Completeness is the percentage of data, which meets the established acceptance criteria referenced in Section 6.0. ATL's goal is to achieve at least 95% completeness for both normal turn-around-time (TAT) and rush TAT data. Meeting the method specification outlined in each SOP prior to analyzing project samples is our means of achieving this goal.

#### **4.1.5 Comparability**

Comparability is the confidence with which one data set may be compared to another. The objective for this QA/QC program is to produce data with the greatest possible degree of comparability. Comparability is achieved by using standard analytical methods, reporting data in standard units, and using standard and comprehensive reporting formats.

### **4.2 LIMIT OF DETECTION, LIMIT OF QUANTITATION, AND INSTRUMENT CALIBRATION REQUIREMENTS**

#### **4.2.1 Limit of Detection**

The Limit of Detection - LOD (or Method Detection Limit) is a statistically determined value (by Method Detection Limit per 40CFR

Part 136 Appendix B). The LOD must be less than the Limit of Quantitation (LOQ). If the true concentration is below this value, the analyte may not be detected. Each LOD study is repeated at least once per twelve-month period, when a new instrument is installed, when there is a major change in the analytical configuration such as column, detector, sample concentrator, sample loop size, etc. or when there is a major change in the extraction method such as solvent, extraction apparatus, clean-up procedure, etc.

All analytical constituents noted by methods in Section 6.0 are to be reported with a valid and current LOD, but in the case of special request compounds LODs are performed only when a client specifies it to be a project requirement. Special request compounds are reviewed by the Department Managers to determine the cost to the laboratory for additional LOD analyses. The additional value added is then factored into the bid that is submitted to the prospective client.

#### **4.2.2 Limit of Quantitation**

ATL reports down to the Limit of Quantitation - LOQ (or Reporting Limit) which is the lowest concentration contained in a linear calibration.

The LOQ represents a uniform value that can be accurately detected for any particular analyte on each instrument thereby providing consistency for samples analyzed on different instruments. The Reporting Limit is verified by the statistical and analytical LOD studies.

The acceptance criterion for the LOD study is a value of less than the LOQ. Corrective action including raising the LOQ may be performed if the statistically and analytically determined LOD does not meet the stated criterion.

#### **4.2.3 Instrument Calibration**

Analytical instruments are calibrated in accordance with the referenced analytical methods and internal SOPs. The acceptance criteria are summarized in Section 6.0. All specific target analytes are included in the initial and continuing calibrations.

If multi-point calibrations do not meet acceptance criteria stated in the relevant SOPs, an option to narrow the range of the curve either by eliminating the low point or the high point of the curve may be considered providing all project criteria are still met. Otherwise, the entire calibration curve is repeated. Reanalysis of any level of the multi-point calibration in order to meet QC acceptance criteria is not allowed unless there is evidence of an anomaly such as instrument malfunction or an improper load volume. Documentation of the anomaly must accompany the raw data for the Initial Calibration. Elimination of any of the inner levels of the multi-point calibration in order to meet QC acceptance criteria is not allowed.

Records of instrument calibration and records that unambiguously trace the preparation of standards and their use in instrument calibration are maintained for 5 years. Calibration standards are traceable to standard materials.

A second source (or different lot) standard that contains all target compounds, as noted in the Section 6.0 tables, is analyzed after each initial calibration curve to verify that the standards are correct and the calibration is accurate. The acceptance criteria for the independent source recoveries are presented in Section 6.0.

In the case of special request compounds, a second source analysis is performed only when a client specifies it to be a project requirement. Special request compounds are reviewed by the Department Managers to determine the cost to the laboratory for additional second source analyses. The

additional value added is then factored into the bid that is submitted to the prospective client.

Analyte concentrations are determined primarily using the average RF from the initial multi-point calibration.

#### **4.2.4 Retention Time Windows**

The techniques used to establish retention time windows for GC and HPLC analyses vary by method, based on the class of compounds targeted, as well as the instrument specifications (e.g., column type, etc.). Protocol for establishing retention time windows can be found in the method-specific SOPs.

### **4.3 ELEMENTS OF QUALITY CONTROL**

The various types of QC samples are described below. The method specific laboratory QC sample frequency and acceptance criteria may be found in Section 6.0.

#### **4.3.1 Analytical Batch Definition**

For non-extractable methods, samples analyzed during a single 24-hour period along with associated matrix specific laboratory QC samples make up an analytical batch. At a minimum, any analytical batch will include a Laboratory Blank, CCV, LCS and an end check for non-GC/MS methods. Reporting of the batch QC samples varies according to project requirements. The number of field samples included in any one analytical batch is limited to 20.

In the case of samples that require extraction prior to analysis, the sample preparation process defines the batch. At a minimum, the sample preparation batch will include a Laboratory Blank and a Laboratory Control Sample (LCS). The maximum number of samples included within one preparation batch may not exceed 20 in one given day.

#### **4.3.2 Continuing Calibration Verification (CCV)**

A Continuing Calibration Verification (CCV) containing all analytes of concern is performed at the start of each day and, if required, at the start of every 12 or 24 hour clock for GC/MS analyses. GC and HPLC sample analyses are generally bracketed by opening and end check CCVs (TO-4A and TO-10A methods excluded). Mid-batch CCVs are also analyzed as per individual SOP.

The concentration of the CCV is usually near the mid-level of the calibration. The CCV is analyzed at other concentrations within the working range at least once a quarter, or more frequently if specified in an SOP. If the CCV fails to meet the performance criteria then the test is repeated with the same standard (or optionally with a different preparation of the same calibration mix). If the second analysis fails criteria, maintenance should be performed and the test repeated a third time. If the system still fails the calibration verification, a new multi-point calibration curve is performed.

#### **4.3.3 Laboratory Control Spike (LCS)**

Each analytical or extraction batch contains at least one mid-level spike using a second source (or different lot) standard containing all the target analytes or the target analytes required by the method. In the case of non-extracted batches, the LCS is generally analyzed daily prior to sample analysis, but may also serve as an End Check standard. If the stated criteria are not met, the system is checked and the standard reanalyzed. In the event that the criteria cannot be met, the instrument is recalibrated. In the case of extracted LCS, out-of-control recoveries result in data flags since samples cannot be re-extracted.

For work that falls under the scope of the DoD QSM the LCS control limits must be derived from historically generated limits. Marginal Exceedances are also evaluated for the LCS and must be within criteria for the analysis to continue.

#### **4.3.4 Internal Standard (IS)**

For all GC/MS methods an IS blend is introduced into each standard and blank to monitor the stability of the analytical system. The internal standard acceptance criteria vary by method, but for all applicable analyses at ATL, if the internal standards for the blank do not pass the acceptance criteria, the system is inspected and the blank reanalyzed. Analyses are discontinued until the blank meets the internal standard criteria.

#### **4.3.5 Surrogates**

For GC/MS methods and some GC methods, the recovery of the surrogate standard is used to monitor for unusual matrix effects, gross sample processing errors, and to provide a measure of recovery for every sample matrix. The surrogate recovery acceptance criteria vary by method, but for all applicable analyses at ATL, if the surrogate recoveries for the Laboratory Blank do not pass the acceptance criteria, the system is inspected and the blank is reanalyzed. Analyses are discontinued until the blank meets the surrogate recovery criteria.

In some extractable methods, surrogates are added prior to extraction to monitor the efficiency of the extraction process. If the surrogate recoveries are outside acceptance limits, reanalysis occurs. Re-extraction of samples is not possible.

If the surrogate recoveries for a sample are outside the limits, the sample is reanalyzed unless obvious matrix interference is documented. If the surrogate recoveries are within limits in the reanalysis, the second analysis will be reported. If the surrogate

recoveries are out of limits a second time, the initial analysis is reported with a narrative indicating that the acceptance criteria for surrogate recoveries are exceeded. Upon request, the data from the matrix effect confirmation analysis is provided to the client.

For work that falls under the scope of the DoD QSM the Surrogate control limits must be derived from historically generated limits

#### **4.3.6 Laboratory Blank**

A Laboratory Blank is analyzed after any applicable standards and prior to the analysis of project samples. A blank is also analyzed in the event saturation-level concentrations are incurred to demonstrate that contamination does not exist. For methods that involve an extraction, a Laboratory Blank is prepared with each set of no more than 20 samples per method per matrix.

The acceptance criterion for the Laboratory Blank is a result less than the Limit of Quantitation (Reporting Limit). The Laboratory Blank is analyzed immediately after the LCS (non-extractable analysis) or the CCV (extractable analysis) to ensure that both the instrument and extraction process are free from contamination. When samples that are extracted together are analyzed on different analytical clocks, a solvent (instrument) blank is analyzed to demonstrate that the instrument is free from contamination.

For work that falls under the scope of the DoD, the acceptance criteria for the Method Blank is as follows:

No analytes detected at  $\geq \frac{1}{2}$  the RL. For common laboratory contaminants, no analytes detected  $\geq$  the RL. If an analyte in the laboratory blank fails these criteria the associated samples must be reprocessed in another analytical batch unless the analyte resulted in a non-detect. In no sample volume remains for re-analysis, the results will be reported with the appropriate data qualifying code (B flag).

#### **4.3.7 Laboratory Duplicate**

Project samples are analyzed in duplicate on a minimum of 10% of the samples received. For some projects the required frequency is one duplicate analysis per analytical batch. The acceptance criteria for analytical reproducibility generally apply to analytes present at  $\geq 5$  times the Reporting Limit. If the noted criterion is exceeded, the sample is re-analyzed a third time. If acceptable reproducibility is still not obtained, the cause is investigated and the system is brought back to working order. If no problem is found on the system, the data is narrated to note the non-conforming event.

#### **4.3.8 Matrix Spike**

Matrix spiking permanently alters the native concentrations of whole air samples. Therefore, matrix spiking is performed only on samples, such as condensates, submitted as part of a sampling train or on extractable samples provided they are submitted in duplicate for matrix spike and in triplicate for the matrix spike duplicate. When applicable, matrix and matrix duplicate spiking is performed using a subset of target analytes. Recoveries and demonstrated reproducibility values, which do not meet the acceptance criteria, are flagged and explained in the laboratory narrative.

#### **4.3.9 Field QC Samples**

Field blanks, field spikes, and field duplicates are generally treated as normal project samples by the laboratory. The exceptions include methods in which the laboratory at the direction of the client specifically prepares the sample media. To assure consistency it is recommended that certified summa canisters connected to a sampling tee be used for the collection of field duplicate samples.

### **4.4 QUALITY CONTROL PROCEDURES**

#### **4.4.1 Holding Times**

All sample preparation and analysis are to be completed within the method-required holding times. The analytical holding time for a non-extractable method begins the day of sample collection. For extractable methods, the holding time is calculated from the day of sample collection for the extraction process and from the day the extraction process begins for the analytical process.

If holding times are exceeded, a CAR form (Section 3.3.2) is generated, the client is notified, and situation is narrated on the final report.

#### **4.4.2 Confirmation**

GC and HPLC methods do generally not perform quantitative confirmation for air sample analysis. The exception is for analysis of pesticides by SW-846 methodology, in which case, second column confirmation is completed within the method-required holding times.

#### **4.4.3 Standard Materials**

All purchased supplies, reagents, solvents and standards are verified as acceptable and meeting criteria for analysis prior to use. All neat and liquid standards used are traceable to the National Institute of Standards and Technology (NIST) and NIST traceable weights are used to verify balance calibration. Documentation from the manufacturers is maintained to verify each standard. Gaseous standards (which are by nature unable to be quantified on a balance) are verified by accuracy documentation supplied by the manufacturer.

A second source (or different lot) standard is used to confirm the accuracy of primary source calibration standards. Ideally the second source is obtained from a vendor other than that of the primary standard. In the case

of TO-14A/TO-15 a reliable second source vendor may be difficult to find and therefore a different lot standard may be used for this purpose. These standards are used for the laboratory control samples as well. Non-standard and polar TO-14A/TO-15 compounds may be prepared from neat standards. Second source standards for these compounds are either derived from a different vendor or from a different lot if only one vendor exists.

#### **4.4.3.1 Liquid Standards**

Liquid Standards are prepared by dilution from commercial sources. Stock solutions are purchased and stored in a separate refrigerator/freezer. Dilutions to working ranges are prepared using high purity solvents. Solvents are logged into the receiving logbook and the date of arrival is documented. Open solvent containers are stored in a vented, flammables cabinet.

#### **4.4.3.2 Gas Standards**

Gas standards are purchased from a commercial supplier and stored in vendor recommended cylinders using high purity regulators. Standards that are not available in certified blends from commercial suppliers are purchased in neat form. Neat materials are purchased with a purity of at least 96% whenever possible.

Certified gas blends are purchased at parts per million volume (ppmv) levels and diluted into the working range by transfer into 1.0 L or 6.0 L certified evacuated Summa™ canister. The canister is then pressurized to 5.0 psi or 15.0 psi depending on the volume. Alternatively, a high purity flow controller is used to fill a conditioned Tedlar bag with a controlled volume of N<sub>2</sub> or zero air. Neat liquid standards are transferred into the Tedlar bag by injection to achieve the desired concentration. The standard is given sufficient time for equilibration and then is transferred into a

conditioned Summa™ canister and pressurized appropriately to achieve the desired final concentration.

Concentration of the blend is determined using density based calculation:

$$\text{ppbv} = \frac{\text{ng/MW}}{\text{vol(L)}/24.45^*}$$

\* 24.45 is the molar volume of any gas at normalized pressure (1 atmosphere) and temperature (25°C), derived from the ideal gas law ( $PV = nRT$ ), where  $R$  = the universal gas constant.

Once blended, the standard is transferred into a Summa™ canister for long-term storage and stability.

The preparation of working standards, in gaseous or liquid states, is documented in bound standard preparation logbooks. Each standard is given a unique identification number. Additional information including the solvent or standard lot number and stock standard concentration is noted. Each page is signed and dated by the analyst.

#### **4.4.3.3 Reagent Water**

The laboratory uses water to prepare moist Laboratory Blank canister samples, VOST condensate water blanks and water impinger blanks. The volume of water required for these purposes is insignificant. As such, the laboratory relies on high purity HPLC grade bottle reagent water, which is subjected to a constant purge flow of Ultra High Purity Nitrogen. The water is purchase certified and then supported by certifying Laboratory Blank analyses.

#### **4.4.4 Expiration Dates of Standards**

##### **4.4.4.1 Primary Standards**

Primary standards expire according to the manufacturer's expiration date. If the manufacturer does not assign an expiration date, a period of one year from the date of opening is applied. Expiration dates are noted on standard labels. Expired standard materials are either revalidated by comparison with unexpired independently prepared standards, or are discarded. The acceptance criterion for standards revalidation is documented in ATL SOP #33. The newer of the two standards is always used as the primary source.

Expiration dates for laboratory-prepared stock and diluted standards are no later than the expiration date of the stock solution or material.

All efforts are made to obtain the highest purity possible when purchasing neat chemical

standards. The vast majority of neat standards are  $\geq 96\%$  pure. The concentration of material purchased at less than 96% purity is corrected mathematically to assure that dilutions for working standards are accurate.

Neat liquid standards are used until analysis by GC/FID indicates a purity of less than 96% (or less than the stated purity for the exceptions). The date of the purity check is noted on the neat standard vial.

Purity analysis is performed once per year or as needed.

##### **4.4.4.2 Secondary Standards**

Secondary Standards are assigned based on the expiration date of the primary source standard (i.e., no later than), the compounds present, and container type. Typical expiration dates are presented in Exhibit 4.2.

**Exhibit 4.1. Statistical Calculations**

Statistic	Symbol	Formula	Definition	Uses
Mean	$\bar{X}$	$\frac{\left( \sum_{i=1}^n X_i \right)}{n}$	Measure of central tendency	Used to determine average value of measurements
Standard Deviation	S	$\left( \frac{\sum (X_i - \bar{X})^2}{(n-1)} \right)^{1/2}$	Measure of relative scatter of the data	Used in calculating variation of measurements
Relative Standard Deviation	RSD	$(S / \bar{X}) \times 100$	Relative standard deviation, adjusts for magnitude of observations	Used to assess precision for replicate results
Percent Difference	%D	$\frac{X_1 - X_2}{X_1} \times 100$	Measure of the difference of 2 observations	Used to assess accuracy
Relative Percent Difference	RPD	$\left( \frac{(X_1 - X_2)}{(X_1 + X_2) / 2} \right) \times 100$	Measure of variability that adjusts for the magnitude of observations	Used to assess total and analytical precision of duplicate measurements
Percent Recovery	%R	$\left( \frac{X_{\text{meas}}}{X_{\text{true}}} \right) \times 100$	Recovery of spiked compound in pure matrix	Used to assess accuracy
Percent Recovery	%R	$\left( \frac{\text{value of spiked sample} - \text{value of unspiked sample}}{\text{Value of added spike}} \right) \times 100$	Recovery of spiked compound in sample matrix	Used to assess matrix effects and total precision
Correlation Coefficient	r	see SW8000B section 7.5.3		Evaluation of "goodness of fit" of a regression line

X = Observation (concentration)

n = Number of observations

**Exhibit 4.2. Expiration Dates**

Gas Standards Prepared from Certified Cylinders

<u>Compounds</u>	<u>Tedlar Bag Standard</u>	<u>Summa™ Canister</u>
TO-14A/15 List	3 days	3 months
BTEX/TPH	3 days	3 months
Sulfur Compounds > 3000 ppbv	7 days	NA
Sulfur Compounds < 3000 ppbv*	1 day	NA

Gas Standards Prepared from Neat Materials

<u>Compounds</u>	<u>Tedlar Bag Standard</u>	<u>Summa™ Canister</u>
TO-14A/15 Extra Compounds	3 days	6 months
Other Compounds	3 days	6 months

Liquid Manufacturer's Certified Mix and Single Component Standards

The manufacturer's expiration date is used when indicated. If none is supplied, the following expiration dates are applied:

<u>Compound</u>	<u>Expiration</u>
Gases in Liquid	1 month after opening ampule
Other Volatile compounds	6 months after opening ampule
Semivolatile/Pesticides/PCBs	1 year after opening ampule

Liquid Stock Standards Prepared from Neat Materials

<u>Compound</u>	<u>Expiration</u>
Gases in Liquid	1 month
Other Volatile Compounds	6 months
Semivolatile/Pesticides/PCBs	1 year

\* Used for Initial Calibration only

**This page left blank intentionally**

## 5.0 SAMPLE HANDLING

### 5.1 SAMPLING MEDIA AND PRESERVATION REQUIREMENTS

General guidelines regarding sampling media, preservation, and holding time requirements are summarized in Exhibit 5.1. The laboratory first refers to project specific requirements. These requirements can be found in the individual Statement of Work (SOW) or Quality Assurance Program Plan (QAPP). If there are no project specific guidelines, the lab uses the criteria presented in Exhibit 5.1.

***Disclaimer:** ATL assumes no real or implied responsibility or liability for client-related field sampling and shipping activities. It is the responsibility of the individual client to ensure that referenced methodologies are followed with respect to sample collection and shipment to the laboratory. Air sampling media and equipment should only be used by experienced field engineers. It is the ultimate responsibility of the client to be knowledgeable both in sample preservation requirements as well as relevant State, Federal, and International shipping requirements. Any time a chemical substance is collected using ATL media, the client bears sole responsibility to understand and abide by the laws involving shipment of potentially hazardous substances by common carrier.*

#### 5.1.1 Sample Containers

Items provided by the laboratory include:

- Sampling media such as Tenax®, Anasorb®-747, charcoal traps, TO-17 tubes, Summa™ polished canisters, PAC 250s, Tedlar bags, PUF/XAD and DNPH impinger solution
- Chain-of-custody forms
- Sampling labels
- Chemical ice packs
- Shipping containers
- Custody Seals (per client request)

- Sample Acceptance Policy

Air sampling media prepared by the laboratory for field use must be certified for cleanliness. Tenax®, Anasorb®-747, charcoal traps, TO-17 tubes, PUF/XAD and DNPH impinger solutions are certified for each preparation batch. The canister cleaning process is certified on a 10% minimum frequency basis. Individually certified canisters are also available per specific client request.

#### 5.1.1.1 Summa™ Canisters

The Support Services Department has dedicated approximately 2600 ft<sup>2</sup> for canister cleaning and certification functions. Approximately 200 canisters can be cleaned daily and up to 120 canisters individually certified daily. This area is also sufficient for storage of approximately 600 canisters and the entire in-house inventory of flow controllers (see Table 2.2).

When canisters are first purchased they are all cleaned, certified and verified as meeting acceptance criteria prior to use. After that, a minimum of ten percent of all canisters that are cleaned per ATL SOP #7 are certified by GC/MS analysis for TO-14A/TO-15 target compounds. 6.0 L and 1.0 L canisters are certified to be clean to 0.2/0.5/0.8 ppbv for the standard product TO-14A/TO-15 target compound list.

If a canister fails certification, the cleaning process is repeated. The canister is not returned to the inventory until it has passed certification. More information on the preparation and certification of Summa™ canisters can be found in ATL SOP #7.

ATL recommends use of 100% certified canisters for projects that require Low Level and SIM TO-14A/TO-15 analysis. Project Managers document requests of this nature in the Project Profile to assure that all shipped media meet this requirement. Canisters and associated sampling train equipment intended

for projects that are defined as Low Level or SIM are certified at or below the Limit of Quantitation. An increase in the percent of canisters certified is also recommended for projects of a very sensitive nature.

Each ATL canister is barcoded, allowing the history of the canister to be maintained through a canister tracking system. The database keeps information, such as, date shipped, client name, date received, and the analytical work order number.

#### **5.1.1.2 Sorbent Tubes**

Each batch of sorbent tubes is certified using the prescribed analytical methodology which most commonly is either SW-846 5041A/8260B or Modified EPA TO-17. One set of tubes from each preparation batch are stored at  $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$  and then certified by GC/MS analysis. Certification is performed before the media is shipped or used to collect samples. The background level of each target VOC must be less than the project reporting limits. The client may allow an exception to this criterion for common laboratory contaminants such as Methylene Chloride but must provide written documentation to verify acceptance. The client will be notified in advance of sampling if the batch fails and a replacement shipment provided if necessary. Tube certification results are reported with the associated Work Order Comprehensive Validation Package. More information of the preparation and certification of sorbent tube media can be found in ATL SOP #4.

#### **5.1.1.3 Polyurethane Foam (PUF/XAD) Cartridges**

PUF and PUF/XAD cartridges are batch cleaned using a large Soxhlet extraction apparatus per SOP #14. The weekly cleaning capacity is 200 PUF/XAD cartridges and approximately 50 media are available for shipping at any point in time (see Table 2.2). For Method TO-13A, two PUFs from each batch are cleaned and extracted and analyzed

by GC/MS. For Methods TO-4A and TO-10A, one PUF from each batch is extracted and analyzed by GC/ECD. For Method TO-13A XAD media, 20 mLs of the media is extracted and analyzed by GC/MS.

The background level of each target analyte must be less than the project reporting limits. Analysis of the extract is performed typically prior to shipping or at very least within 24 hours of shipping. The client is notified in advance of sampling if the batch fails and a replacement shipment is provided if necessary.

#### **5.1.1.4 2,4-Dinitrophenylhydrazine (DNPH) Impinger Solution and Cartridges**

The DNPH solution is prepared in bulk solution. An aliquot is removed, extracted and analyzed according to the particular method (CARB 430, Method 0011, Compendium Methods TO-5 and TO-11A). The DNPH reagent and the TO-11A media are certified as acceptable when the concentration of each analyte in the certification is below the reporting limit. In the case that contamination is present above the reporting limit; the source must be identified and eliminated prior to shipping of the reagent and the TO-11A media. If the contamination can not be eliminated, the presence of any aldehyde necessitates a call to the client. The data user, particularly in the case of source testing, may tolerate concentrations of target aldehydes near the reporting limit. Certification is completed before solution is shipped to the field. Per client request, results are shipped with the media or faxed to the site and are also kept on file in the laboratory.

More information of the preparation and certification of DNPH media can be found in ATL SOP #62.

**5.2 SAMPLE COLLECTION PROCEDURES  
 – FIELD GUIDELINES**

**5.2.1 Information for Canister Sampling**

Air Toxics Ltd. provides a technical “how to” guide on canister sampling. The guide includes the following information:

<b><i>AIR TOXICS'</i></b>	
<b>GUIDE TO AIR SAMPLING AND ANALYSIS</b>	
<b>Canisters and Tedlar Bags</b>	
<b>Sixth Edition</b>	
<b>1.0 Introduction</b>	
1.1 Whole Air Sampling of VOCs	
1.2 Choosing Between Canisters and Tedlar Bags	
<b>2.0 Canisters and Associated Media</b>	
2.1 Introduction to Canisters	
2.2 Associated Canister Hardware	
<b>3.0 Sampling with Cansiters</b>	
3.1 Grab Samples	
3.2 Integrated Samples	
<b>4.0 Sampling with Tedlar Bags</b>	
4.1 Introduction to Tedlar Bags	
4.2 Tedlar Bag Sampling	
<b>5.0 Special Consideration Sampling</b>	
5.1 Special Sampling Configurations	
5.2 Considerations for Sampling at Altitude	
5.3 Considerations for Soil Gas/Landfill Gas Sampling	
<b>Tables</b>	
1.2	Comparison of Canisters to Tedlar Bags
2.2.3	Fill Times for Canisters
3.2.3	Flow rates For Selected Sampling Intervals
3.2.4	Relationship Between Final Canister Vacuum, Volume Sampled and Dilution Factor

**5.2.2 Information for Sorbent Tube Sampling**

Contents of the guide include:

<b><i>AIR TOXICS'</i></b>	
<b>GUIDE TO AIR SAMPLING AND ANALYSIS</b>	
<b>Sorbents and Solutions</b>	
<b>Second Edition</b>	
<b>1.0 Introduction</b>	
<b>2.0 Sorbent Sampling</b>	
2.1 Considerations for Sorbent Sampling	
2.2 Method Specific Sampling Instructions	
EPA Method TO-4A	
EPA Method TO-10A	
EPA Method TO-13A High Volume	
EPA Method TO-13A Low Volume	
EPA Method 0010/8270C by MM5 Train	
EPA Method TO-17	
EPA Method VOST 0030/5041A	
NIOSH 5515	
<b>3.0 Solution Sampling</b>	
3.1 Method Specific Sampling Instructions	
EPA Method TO-5	
EPA Method TO-11A	
CARB 430 Method	
EPA Methods 0011	
Air Toxics Ltd. Method - Siloxanes	
<b>4.0 Filter Sampling</b>	
4.1 Method Specific Sampling Instructions	
EPA Method PM10 & TSP	

The information provided in ATL's Sampling Guide is meant to serve only as general guidelines. In all cases, field sampling personnel are ultimately responsible for having expertise and knowledge in air sampling methodology sufficient to ensure that the defensibility of the data will not be compromised due to deficiencies in field sampling, handling or transportation.

### **5.3 SAMPLE RECEIVING PROCEDURES**

Upon arrival at the laboratory, samples are received and inspected following Air Toxics' sample acceptance criteria as outlined in SOP #50. The SOP establishes specific guidelines for sample acceptance, which are generally accepted practices under EPA, AFCEE, USACE, Navy, and NELAP protocols. When samples do not meet the established guidelines, discrepancies are documented and the client is notified. Samples are noted in the individual Work Order and discrepancies noted in the Laboratory Narrative portion of the sample report.

#### **5.3.1 Sample Acceptance Policy**

Samples received by Air Toxics Ltd. must be relinquished following standard EPA approved guidelines. These include full and complete Chain of Custody (COC) documentation indicating:

- Unique sample name
- Location, date, and time of collection
- Canister Number
- Collector's name
- Preservation type (if applicable)
- Matrix
- Any special remarks

The COC form (Exhibit 5.2 and 5.2b) must be filled out in ink and indicate proper preservation and use of sample container specified by the method. Each sample should be labeled with unique, durable, and indelible identification and must be of adequate volume

for the tests requested. Never affix a label directly on a Summa™ canister. A tag is attached to each canister for this purpose.

Proper, full, and complete inspection and documentation will be performed upon laboratory receipt in the following areas:

- Evidence of container's physical damage
- Status of the container's custody seal
- Presence or absence of a COC form
- Incomplete or incorrect COC form
- Number of samples
- Name of each sample
- Sample collection date/time
- Name of the Project Manager
- Canister ID (if applicable)
- Preservation type (if applicable)
- Sample type (canister, XAD, DNPH etc.)
- Sample tag information complete
- Temperature (if applicable)
- Pressure (canisters)
- Presence of unlabeled samples
- Presence of mis-labeled samples
- Presence of unused media
- Method required trip blanks, field blanks, equipment blanks, field duplicates, or field spikes

Any sample discrepancies against the above criteria are documented on the Sample Discrepancy Form (Exhibit 5.3), and communicated to the client via Electronic Sample Receipt Confirmation within 1 day of sample receipt. The client is contacted by the Project Manager for discrepancies of a more serious nature, e.g.,

- Chain of Custody Record was not received with sample(s).
- Analysis method(s) is(are) not specified.
- Sample(s) received out of holding time.
- Sample container (Tube/VOA vial) was received broken.
- Canister sample received at >15" Hg (not identified as a Trip/Field Blank).
- Tedlar Bag received leaking.

- Tedlar Bag received flat.
- Tedlar bag / canister received emitting a strong odor (sample cannot be analyzed).

Documentation of client notification is included on the form along with any instructions from the client on how to proceed. Project Managers complete this section and return the form to the Receiving group to complete the login process. The form is archived in the Work Order folder. Whenever there is any uncertainty of how the laboratory is to proceed or when the desired method is unclear, the Receiving staff places the Login process ON HOLD and delivers the Work Order file to a Project Manager for follow-up. The Project Manager contacts the client to clarify the situation. Phone calls between the Project Manager and the client are documented in the Project Management Software. The phone contact and client instructions to resolve the issue are logged into the database and a hardcopy report is placed in the Work Order folder. The folder is then returned to the Receiving team to complete the Login process. Air bills, packing lists, Chain of Custody records, and any other documentation that may accompany the samples are placed in the Work Order folder.

Laboratory malfunctions occurring during/after sample receipt are documented via the laboratory Corrective Action system. Examples of receiving problems, which would necessitate a Corrective Action Request, include:

- Hold time expired due to laboratory error.
- Canister sample pressurized with wrong type of gas.
- Sample placed On Hold was released in error.
- Sample logged in for incorrect analysis method.
- DANGER tag was not affixed to an odiferous canister sample before sending to the lab.

- Canister was released and cleaned before second analysis method was run.
- Canister valve was left open following pressurization. Sample vented to ambient.

### **5.3.2 The Sample Receipt Confirmation**

When a Work Order is completed, Sample Receipt Confirmation (SRC) is sent to the client to confirm receipt of samples. A fax is sent if no email address is available. The Sample Receipt Confirmation has six sections:

*Section 1 Introduction Page (not available if faxed)*

*Section 2 Cover page with discrepancies noted*

*Section 3 Sample Receipt Summary (sample names, etc.)*

*Section 4 Copy of Chain of Custody*

*Section 5 Reporting template showing referenced method, target*

*compound list, and reporting limits*

*Section 6 Media outstanding (if relevant)*

Discrepancies are noted on the cover page using a template of pre-approved statements. The QA Department is responsible for maintaining the approved template. Receiving staff electronically copy relevant statements from the template and onto the SRC cover page. Typical statements include:

- NELAC Chapter 5 specifies that a legal Chain of Custody must accompany samples when they arrive at the laboratory. In this case a chain of custody was not received with the samples. The discrepancy was noted in the Login email.
- The Chain of Custody (COC) form was not relinquished properly. A <signature OR date> was not provided.
- Samples were received past the recommended hold time of \_\_\_\_\_ days. Analysis proceeded.
- The sample collection date was incomplete on the Chain of Custody

(COC) for samples(s) <insert names>. The client was contacted and a date of <enter date> was provided.

- The Tedlar bag for sample \_\_\_\_\_ was received flat. The client was notified that analysis was not possible.
- A Temperature Blank was included with the shipment. Temperature was measured and was not within  $4\pm 2$  °C. Coolant in the form of ice/blue ice was present. Analysis proceeded.

### **5.3.3 The Work Order Folder**

A folder is created during the Login process to hold all relevant documents. The folder is labeled with the unique Work Order number, client name and analysis. One folder for each desired analysis is created so that laboratory analyses can be efficiently handled as separate processes. The folder contains the following receiving/login documents:

- Original COC record, airbill, and any other packing documents
- Sample Receipt Summary with individual field sample names, dates of collection and project reference
- Specific method cited, and a copy of the reporting target compound template for review
- Login Review Checklist
- The Sample Discrepancy Report (attached electronically)
- Copy of the Project Management Project Profile with associated special analysis and reporting requirements
- The Receiving Report (for ATL media only)

- ATL Shipment Report (attached electronically if shipping charges apply)
- Copy of any approved Project Requirement Tables generated after the bid has been won

The folder is passed to the analytical teams after Login, and follows the same process stream as the samples. All original documents generated during the processing of the samples are filed in this folder. The unique Work Order file makes archival and retrieval of evidentiary and custodial documents easier. The majority of analytical documentation is archived electronically. Documentation that remains in hard copy form includes:

- COC
- Data Review Checklist
- Corrective Action Requests
- Scan Packets (run logs, spectral defenses, manual integrations etc.)
- Phone contacts and e-mails
- Fed-Ex/UPS air bill/freight bill
- GC/FID screening results

Alternatively, the Work Order folder is placed in a bar coded storage box for long-term storage. The storage boxes are either stored on-site or Work Order inventory of each box is taken prior to offsite storage and maintained along with the bar code address. A private storage company archives the boxes by barcode and provides one-day retrieval service upon request.

### **5.4 SAMPLE TRACKING PROCEDURES**

After samples have been inspected, they are given a unique tracking number and logged into an electronic sample receiving database. The tracking number consists of the year and month plus a sequential Work Order number. As an example, the first set of samples received in July, 2004 would have the format:

#### **0407001**

If this set of samples consisted of eight individual samples, then each sample is identified by a consecutive postscript such as:

#### **0407001-01A through 08A**

If more than one analysis is requested for the samples, an alphabetic designation is given to each analysis sample set:

#### **0407001A-01A TO-15 0407001B-01A TO-3**

Laboratory assigned duplicates are designated using a double postscript such as:

#### **0407001-01AA**

A more detailed discussion of the sample receiving function is given in ATL SOP #50. The laboratory processes thousands of samples each month divided into hundreds of individual Work Orders. An efficient user-friendly database is critical in keeping track of each individual sample, monitoring hold times, monitoring due dates, and scheduling analyses. In addition, most air projects have specific target compound lists, reporting limit requirements, quality assurance requirements, analysis requirements, and data submission requirements. Relevant project information is immediately available as each processing step occurs. The ultimate goal of the ATL sample-processing system is to deliver what the customer wants the first time. Report re-issues and sample re-analyses are monitored and kept to a minimum. In order to meet the quality objective (customer satisfaction), every team member has access to information describing what the customer has requested.

The sample tracking database consists of a variable number of data fields sufficient to store project and sample batch information. The users can then query any field in the database. Each department creates work lists from the database and inputs relevant

information (e.g., completion dates, etc.) throughout the day.

The database resides on a secured network server equipped with a daily-automated back up system. Multiple PCs are available to each team in their respective work areas. Access privileges are defined and maintained by the IT team. The database is designed such that Work Order status can be determined at any point in time. The 'status' field is updated each time the work progresses to a new stage in its processing. Status data include:

- Client Services
- Extractions
- Log-in
- Lab Bins
- Individual Instrument Assignment
- Data Review
- QA
- FAX
- EDD Generation
- Final Report
- Financial Hold
- Filed

Complete documentation of sample processing is maintained in the database. Each team completes relevant portions of the database as work is finished. Selected information includes:

## SAMPLE TRACKING FIELDS

**Work Order number**  
**Client Services contact**  
**Date received**  
**Client name**  
**Project name**  
**Project ID number**  
**#Samples**  
**Date sampled**  
**#Lab dups**  
**#Sample holds**  
**Container type**  
**Expiration date**  
**Method specific analysis code**  
**Date promised**  
**Rush turn**  
**24 hour clock**  
**Screen done**  
**Date receiving done**  
**Receiving analyst initials**  
**Log-in date**  
**Log-in analyst initials**  
**Date analysis done**  
**Date reported**  
**Bench analyst initials**  
**Date of final report**  
**CVP due date**  
**Date CVP completed**  
**Date CVP shipped**  
**CVP analyst initials**  
**EDD due date**  
**EDD completed date**  
**Date EDD shipped**  
**EDD analyst initials**  
**Reissue due date**  
**Reissue reason**  
**Time Due**

The electronic database is used to document and ensure that analytical hold times, reporting requirements, and project specific QC requirements are met. The database is used by the user to provide project specific activity reports and status of incomplete work. Users may query the database and easily produce a printed report. The sample database is the key

to efficient information transfer and, as such, is a critical tool to meet the quality objective.

### 5.5 INTERNAL SAMPLE CUSTODY AND STORAGE PROCEDURES

The chain-of-custody for samples is documented from time of receipt until time of disposal. Internal sample chain-of-custody documentation consists of:

- Storage area logbooks
- Instrument run logs
- Raw analytical data for samples, calibrations and QC checks

The samples are stored in the custody cage, in a secure refrigerator, or in the event of late delivery in the receiving section until the next morning. The Receiving staff or pressurization personnel log the samples into the Internal Sample or Extractable Sample Tracking Logbook in the storage area.

Samples are tracked in/out of the limited access area by initials, date, and time. All staff members have access to the storage areas and all members are trained on proper custody documentation in the logs. Logbook protocol training is mandatory for all staff. The training and documentation of training is handled by the QA team. The QA team checks the Logbook Review Checksheet monthly to ensure that the analysts have reviewed their logbooks on a timely basis.

### 5.6 SAMPLE DISPOSAL

Samples are released for disposal upon satisfactory completion of analysis unless prior contractual arrangements have been made. The release of samples is documented in the Internal Sample Tracking Log via a "Released" stamp that includes the date and initials of the person who releases the sample for disposal. Samples are released following the procedures outlined in ATL's SOP #63.

Sample disposal varies based on the sampling media. Whole air samples are vented through a charcoal scrubber, while liquid (i.e., solvent and water) samples are disposed of according to the procedures noted in ATL's Chemical Hygiene Plan.

## **5.7 SUBCONTRACTING**

Air Toxics Limited subcontracts samples on an infrequent basis. Subcontracting is generally performed for contractual reasons in fields of testing which the laboratory does not perform. In the event that subcontracting is necessary, the client, working with the Sales or Project Manager, selects a suitable subcontract laboratory that meets the project specified certification criteria. Work that falls under the scope of NELAC accreditation shall be placed with a laboratory accredited under NELAP for the tests to be performed or with a laboratory that meets applicable statutory and regulatory requirements for performing the tests and submitting the results of tests performed. The laboratory performing the subcontracted work shall be indicated in the final report and non-NELAP accredited work shall be clearly identified. If the project has no criteria, then the client may choose to select a subcontract laboratory based on state certification in the desired field of testing. If subcontracting samples is necessary for an analysis that we normally perform, the Project Manager shall advise the client of the arrangement in writing and, when possible, gain the approval of the client, preferably in writing.

Work that falls under the scope of the DoD shall be placed with a laboratory that is DoD accredited and meets the DoD QSM requirements for the tests to be performed. In addition, the subcontracted laboratory must receive project-specific approval from the DoD client before any samples are analyzed. More information for subcontracting samples can be found in ATL SOP #90.

**Exhibit 5.1.**

**Requirements for Containers, Preservation Techniques, and Holding Times**

Method	Parameter	Type	Container	Preservation	Extraction Holding Time	Analytical Holding Time
VOST 5041A/8260B	VOCs	GC/MS	Sorbent Tube	4°C	NA	14 days
TO-14A, TO-3 & CARB 410A	BTEX/TPH	GC/FID/PID	Summa Canister Tedlar Bag	NA NA	NA NA	30 days 3 days
TO-4A & TO-10A	Pesticides PCBs	GC/ECD	PUF	4°C	7 days	40 days
TO-5 & CARB 430	Aldehydes & Ketones	HPLC/UV	DNPH Impinger	4°C	7 days	30 days
TO-11A	Aldehydes & Ketones	HPLC/UV	Sep-PAK	4°C	14 days	30 days
TO-12	NMOC	GC/FID	Summa Canister Tedlar Bag	NA NA	NA NA	30 days 3 days
TO-13A/ 8270	PAHs/ Semivolatiles	GC/MS	XAD/PUF	4°C	7 days	40 days
TO-14A/15	VOCs	GC/MS	Summa Canister Tedlar Bag	NA NA	NA NA	30 days 3 days
TO-17	VOCs	GC/MS	Sorbent Tube	4°C	NA	30 days
ASTM D1946	Fixed Gases CH <sub>4</sub> , C <sub>2</sub> <sup>+</sup>	GC/TCD/FID	Summa Canister Tedlar Bag	NA NA	NA NA	30 days 3 days
ASTM D1945	Fixed & Natural Gases	GC/TCD/FID	Summa Canister Tedlar Bag	NA NA	NA NA	30 days 3 days
ASTM D5504	Sulfur Gases	GC/SCD	Tedlar Bag	NA	NA	24 hours
Method 0011	Aldehydes & Ketones	HPLC/UV	DNPH Impinger	4°C	7 days	30 days
PM10/TSP	Particulate Matter	Analytical Balance	Quartz Filter	59°F – 86°F RH < 45%	NA	14 days

**Exhibit 5.2 General Chain-of-Custody**



**AIR TOXICS LTD.**  
 AN ENVIRONMENTAL ANALYTICAL LABORATORY

**CHAIN-OF-CUSTODY RECORD**

**Sample Transportation Notice**  
 Relinquishing signature on this document indicates that sample is being shipped in compliance with all applicable local, State, Federal, national, and international laws, regulations and ordinances of any kind. Air Toxics Limited assumes no liability with respect to the collection, handling or shipping of these samples. Relinquishing signature also indicates agreement to hold harmless, defend, and indemnify Air Toxics Limited against any claim, demand, or action, of any kind, related to the collection, handling, or shipping of samples. D.O.T. Hotline (800) 467-4922.

180 BLUE RAVINE ROAD, SUITE B  
 FOLSOM, CA 95630-4719  
 (916) 985-1000 FAX (916) 985-1020

Contact Person \_\_\_\_\_  
 Company \_\_\_\_\_ Email \_\_\_\_\_  
 Address \_\_\_\_\_ City \_\_\_\_\_ State \_\_\_\_\_ Zip \_\_\_\_\_  
 Phone \_\_\_\_\_ Fax \_\_\_\_\_

**Project Info:**  
 P.O. # \_\_\_\_\_  
 Project # \_\_\_\_\_  
 Project Name \_\_\_\_\_

**Turn Around Time:**  
 Normal  
 Rush  
 Date: \_\_\_\_\_  
 Pressurization Gas: N<sub>2</sub> He  
specify

Lab I.D.	Field Sample I.D. (Location)	Can#	Date	Time	Analyses Requested	Canister Pressure/Vacuum			
						Initial	Final	Receipt Final (end)	
Relinquished by: (signature) _____ Date/Time _____						Received by: (signature) _____ Date/Time _____			
Relinquished by: (signature) _____ Date/Time _____						Received by: (signature) _____ Date/Time _____			
Relinquished by: (signature) _____ Date/Time _____						Received by: (signature) _____ Date/Time _____			
Notes:									
Shipper Name _____		Air Bill # _____		Temp (°C) _____		Condition _____		Customer Seals Intact? Yes No None	
Lab Use Only		Work Order # _____							



### Exhibit 5.3 Sample Discrepancy Form

#### Sample Discrepancy Report

**Identification**

Initiated By: \_\_\_\_\_ Project ID: \_\_\_\_\_ PM: \_\_\_\_\_ Date: \_\_\_\_\_ Discrepancy Type:  1.  2.  3.

Workorder(s) affected: \_\_\_\_\_ Sample(s) affected: \_\_\_\_\_

**1. Sample Receipt Discrepancies**

**Narration Not Required:**

- 1.1.  Sample container (cartridge/tube/VOA vial) was received broken, however sample was intact.
- 1.2.  No brass cap on canister.
- 1.3.  Date of Collection noted on first sample, but no arrow down to indicate all samples.

**Notify Lab for further determination:**

- 1.4.  Tedlar bag received with minimal volume.

Initials: \_\_\_\_\_ Date: \_\_\_\_\_

**Narration Required In Lab Narrative and Sample Confirmation:**

- 1.5.  COC was not filled out in Ink.
- 1.6.  COC Improperly relinquished / received.
- 1.7.  Sample tags / can numbers do not match the COC.
- 1.8.  Sample date  error /  missing on COC but noted on sample tag (check one).
- 1.9.  Custody Seal on the outside of the container was  broken /  Improperly placed (check one).
- 1.10.  ID-none on the sample Tag/Blank
- 1.11.  Other (describe below).

Describe the Discrepancy: \_\_\_\_\_

**2. Sample Receipt/Screening Discrepancies requiring PM notification**

*Document on Cover Page of Sample Receipt Confirmation and in Receiving Notes of Lab Narrative*

**If Section II. is filled out PM must be notified within 24 hrs of initiation**

- |   |   |
|---|---|
| <ul style="list-style-type: none"> <li>2.1. <input type="checkbox"/> COC was not received with samples.</li> <li>2.2. <input type="checkbox"/> Analysis method(s) is <input type="checkbox"/> not specified / <input type="checkbox"/> incorrectly specified (check one) on the COC.</li> <li>2.3. <input type="checkbox"/> Incorrect sampling media / container for analysis requested.</li> <li>2.4. <input type="checkbox"/> Number of samples on the COC does not match the number of samples that were received.</li> <li>2.5. <input type="checkbox"/> Samples were received expired.</li> <li>2.6. <input type="checkbox"/> Sampling date (time for sulfur) is not documented for <input type="checkbox"/> some / <input type="checkbox"/> any samples (check one).</li> <li>2.7. <input type="checkbox"/> Sample received with amount of H<sub>2</sub>O in the Tedlar Bag.</li> <li>2.8. <input type="checkbox"/> Sample cannot be analyzed. Container was <input type="checkbox"/> received broken / <input type="checkbox"/> leaking / <input type="checkbox"/> flat / <input type="checkbox"/> defective.</li> <li>2.9. <input type="checkbox"/> Tedlar bag / canister received emitting a strong odor; Sample <input type="checkbox"/> can / <input type="checkbox"/> cannot (check one) be analyzed.</li> <li>2.10. <input type="checkbox"/> Tedlar Bag for Sulfur analysis has metal filling.</li> <li>2.11. <input type="checkbox"/> Environmental Supply Company valves</li> <li>2.12. <input type="checkbox"/> Sorbent samples-sampling volume was not provided</li> </ul> | <ul style="list-style-type: none"> <li>2.13. <input type="checkbox"/> Flow controller used – canister samples received at ambient or under pressure.</li> <li>2.14. <input type="checkbox"/> Canister was at ambient pressure at time of pressurization and (check all that apply):           <ul style="list-style-type: none"> <li><input type="checkbox"/> Canister failed leak check on two manifolds,</li> <li><input type="checkbox"/> Canister valve was open,</li> <li><input type="checkbox"/> Brass nut was loose/not present.</li> <li><input type="checkbox"/> Sample can be analyzed</li> <li><input type="checkbox"/> Cannot be analyzed</li> </ul> </li> <li>2.15. <input type="checkbox"/> Canister sample received with a vacuum difference &gt;5.0"Hg between the receipt vac. And the final vac. reported on the COC, indicating loss of vacuum.</li> <li>2.16. <input type="checkbox"/> Canister sample received at &gt;15"Hg (<u>not</u> identified as a Trip/Field Blank).</li> <li>2.17. <input type="checkbox"/> Canister Trip Blank received at low vacuum (&lt; 25"Hg).</li> <li>2.18. <input type="checkbox"/> Sorbent Sample received outside method required temperature of 2°C to 6°C; <input type="checkbox"/> ice / <input type="checkbox"/> blue ice (check one) was present. A temp. Blank <input type="checkbox"/> was / <input type="checkbox"/> was not present (check one).</li> <li>2.19. <input type="checkbox"/> Other (describe below)</li> </ul> |
|---|---|

Initials: \_\_\_\_\_ Date: \_\_\_\_\_ Notify Receiving:  Notify PM:

Describe the Discrepancy: \_\_\_\_\_

Exhibit 5.3 Sample Discrepancy Form (Continued)

**3. Lab Discrepancies requiring Team Leader/PM notification**

Document in Analytical Notes of Lab Narrative

**If Section III. is filled out PM must be notified within 24 hrs of initiation**

- |  |  |
|--|--|
| 3.1. <input type="checkbox"/> Tedlar Bag found to be leaking at the time of analysis; sample <input type="checkbox"/> can / <input type="checkbox"/> cannot (check one) be analyzed. | 3.8. <input type="checkbox"/> Sample loss due to instrument malfunction / broken glassware.                |
| 3.2. <input type="checkbox"/> Tedlar Bag found to be flat/low volume; sample cannot be analyzed.   | 3.7. <input type="checkbox"/> Low/high surrogate recoveries noted in QC/sample(s) for extractable samples. |
| 3.3. <input type="checkbox"/> Sulfur samples received with insufficient time to analyze prior to expiration.   | 3.8. <input type="checkbox"/> Reporting Limit was raised.  |
| 3.4. <input type="checkbox"/> Canister found to be leaking at the time of analysis.  | 3.9. <input type="checkbox"/> Post weight > Pre weight in field/lab Blank for PM10/TSP samples.            |
| 3.5. <input type="checkbox"/> VOST tube saturated; bag dilution necessary.   | 3.10. <input type="checkbox"/> Other (describe below).   |

Initials: \_\_\_\_\_ Date: \_\_\_\_\_ Notify Receiving:  Notify PM:

Team Lead Initials: \_\_\_\_\_ Date: \_\_\_\_\_

Describe the Discrepancy: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

How Does this Affect Client: \_\_\_\_\_  
\_\_\_\_\_  
\_\_\_\_\_

**Project Manager Use Only**

**Project Manager Notification**

**Action:**

- It is not necessary to notify the client. Narrate the discrepancy in Receiving Notes/Analytical Notes of Lab Narrative.

PM Initials: \_\_\_\_\_ Date: \_\_\_\_\_

- Client notification required. See attached client contact / email, or comments below:

Client Notification:

PM Initials: \_\_\_\_\_ Person notified: \_\_\_\_\_ Date: \_\_\_\_\_

- Waiting for Client Reply

Comments: \_\_\_\_\_  
\_\_\_\_\_

Notify Lab Name: \_\_\_\_\_ Date: \_\_\_\_\_ Notify Receiving:

- Additional notifications attached.

**Additional Comments:**

**This page left blank intentionally.**

## 6.0 ANALYTICAL METHODS AND PROCEDURES

This section contains subsections for each analytical procedure. Each subsection contains the following information:

- A brief method description
- Laboratory variances to Compendium and SW-846 methodologies
- A table of LOQs and QC acceptance criteria
- A table of calibration procedures and QC procedures

This Quality Manual references methods in a general manner. The specific revisions used by the laboratory can be found in the method-specific SOPs.

### 6.1 VOST SW-846 5041A/8260B

This method involves GC/MS full scan analysis of volatile organic compounds in air samples collected on Tenax/Charcoal (VOST) cartridges. Samples are collected using SW-846 Method 0030/0031 Volatile Organic Sampling Train (VOST) protocols. The VOST cartridges are thermally desorbed by heating and purging with Ultra High Purity Helium. The resulting gaseous effluent is then bubbled

through 5 ml of organic free reagent grade water and trapped on the sorbent trap of the purge and trap system. The trap is then thermally desorbed for GC/MS analysis. For condensate analysis, a 5 ml aliquot of condensate sample is placed directly in the sparge vessel of the purge and trap (P&T) system and analyzed in a similar manner.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6-1.1. Summary of Method Modifications**

<b>Requirement</b>	<b>EPA Method 5041A/8260B</b>	<b>Air Toxics Ltd. Modifications</b>
Method Blank	Cartridges from the same media batches as the samples.	Media batch is certified prior to use in the field. Method Blank is from a different batch unless requested by the client.
Connection between thermal desorption apparatus & purge vessel.	PTFE 1/16" Teflon tubing.	Heated, 1/16" silica lined stainless steel tubing.
Calibration Criteria for non-CCCs.	RSD ≤ 15 % for all non-CCCs.	RSD ≤ 30 % for Acetone, Bromoform, Vinyl Acetate, Bromomethane, Chloromethane, 1,1,2,2-Tetrachloroethane, & 1,2,3-Trichloropropane.

Table 6.1.2 SW-846 Modified Method 5041A Standard Analyte List

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
1,1,1-Trichloroethane	10	15	70 – 130	-
1,1,1,2-Tetrachloroethane	10	15	70 – 130	-
1,1,2,2-Tetrachloroethane – SPCC	10	30	70 – 130	RF > 0.30
1,1,2-Trichloroethane	10	15	70 – 130	-
1,1-Dichloroethane – SPCC	10	15	70 – 130	RF > 0.10
1,1-Dichloroethene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
1,1-Dichloropropene	10	15	70 – 130	-
1,2,3-Trichlorobenzene	50	15	70 – 130	-
1,2,3-Trichloropropane	10	30	70 – 130	-
1,2,4-Trichlorobenzene	50	15	70 – 130	-
1,2,4-Trimethylbenzene	10	15	70 – 130	-
1,2-Dibromo-3-chloropropane	50	15	70 – 130	-
1,2-Dichlorobenzene	10	15	70 – 130	-
1,2-Dichloroethane	10	15	70 – 130	-
1,2-Dichloropropane – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
1,3,5-Trimethylbenzene	10	15	70 – 130	-
1,3-Butadiene <sup>1</sup>	50	30	50 – 150	-
1,3-Dichlorobenzene	10	15	70 – 130	-
1,3-Dichloropropane	10	15	70 – 130	-
1,4-Dichlorobenzene	10	15	70 – 130	-
2,2-Dichloropropane	50	15	70 – 130	-
2-Butanone <sup>2</sup>	50	30	50 – 150	-
2-Chloropropane	10	15	70 – 130	-
2-Chlorotoluene	10	15	70 – 130	-
2-Hexanone <sup>2</sup>	50	30	50 – 150	-
3-Chloropropene	50	15	70 – 130	-
4-Chlorotoluene	10	15	70 – 130	-
4-Methyl-2-pentanone <sup>2</sup>	50	30	50 – 150	-
Acetone <sup>2</sup>	50	30	50 – 150	-
Acrylonitrile	50	15	70 – 130	-
Benzene	10	15	70 – 130	-
Bromobenzene	10	15	70 – 130	-
Bromochloromethane	10	15	70 – 130	-
Bromodichloromethane	10	15	70 – 130	-
Bromoform – SPCC	10	30	70 – 130	RF > 0.10
Bromomethane <sup>2</sup>	10	30	50 – 150	-
Butylbenzene	10	15	70 – 130	-
Carbon Disulfide	10	15	70 – 130	-
Carbon Tetrachloride	10	15	70 – 130	-

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Chlorobenzene – SPCC	10	15	70 – 130	RF > 0.30
Chloroethane	10	15	50 – 150	-
Chloroform – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
Chloromethane – SPCC	10	30	50 – 150	RF > 0.10
cis-1,2-Dichloroethene	10	15	70 – 130	-
cis-1,3-Dichloropropene	10	15	70 – 130	-
cis-1,4-Dichloro-2-butene	50	15	70 – 130	-
Cumene	10	15	70 – 130	-
Dibromochloromethane	10	15	70 – 130	-
Dibromomethane	10	15	70 – 130	-
Ethylbenzene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
Ethylene Dibromide	10	15	70 – 130	-
Freon 11	10	15	70 – 130	-
Freon 12	10	15	50 – 150	-
Freon 113	10	15	70 – 130	-
Hexachlorobutadiene	50	15	70 – 130	-
Hexane	10	15	70 – 130	-
Iodomethane	50	15	70 – 130	-
Methylene Chloride	10	15	70 – 130	-
Methyl t-butyl ether (MTBE)	10	30	70 – 130	-
Naphthalene	50	15	70 – 130	-
m,p-Xylene	10	15	70 – 130	-
o-Xylene	10	15	70 – 130	-
p-Cymene	10	15	70 – 130	-
Propylbenzene	10	15	70 – 130	-
sec-Butylbenzene	10	15	70 – 130	-
Styrene	10	15	70 – 130	-
tert-Butylbenzene	10	15	70 – 130	-
Tetrachloroethene	10	15	70 – 130	-
Toluene – CCC	10	30	70 – 130	%D ≤ 25% VOST tubes; ≤20% condensates
trans-1,2-Dichloroethene	10	15	70 – 130	-
trans-1,3-Dichloropropene	10	15	70 – 130	-
trans-1,4-Dichloro-2-butene	50	15	70 – 130	-
Trichloroethene	10	15	70 – 130	-
Vinyl Acetate <sup>1,2</sup>	50	30	50 – 150	-
Vinyl Bromide <sup>1</sup> (Bromoethene)	50	30	50 – 150	-
Vinyl Chloride – CCC	10	30	50 – 150	%D ≤ 25% VOST tubes; ≤20% condensates

<sup>1</sup> Independent source verification check not available for these compounds.

<sup>2</sup> Due to nature of these compounds, recoveries outside of noted limits do not result in re-calibration.

**Table 6.1.3 Matrix Spike/Matrix Spike Duplicate**

Analyte	%R
1,1-Dichloroethene	60 – 140
Benzene	60 – 140
Trichloroethene	60 – 140
Toluene	60 – 140
Chlorobenzene	60 - 140

**Table 6.1.4 Internal Standards**

Analyte	CCV IS (%R)	Sample IS (%R)
1,4-Dichlorobenzene-d <sub>4</sub>	50 – 200	60 – 140
Chlorobenzene-d <sub>5</sub>	50 – 200	60 – 140
Fluorobenzene	50 – 200	60 – 140

**Table 6.1.5 Surrogates**

Analyte	%R
1,2-Dichloroethane-d <sub>4</sub>	70 – 130
4-Bromofluorobenzene	70 – 130
Dibromofluoromethane	70 – 130
Toluene-d <sub>8</sub>	70 – 130

**Table 6.1.6 Summary of Calibration and QC Procedures for SW-846 Modified Method 5041A**

*Note: These criteria are used specifically for the standard list of analytes listed in Table 6-1.2.*

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at the start of every 12-hour clock.	Method 5041A tuning criteria.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	SPCC criteria in Table 6-1.2, CCC and non-CCC compound criteria in Table 6.1.2.	Correct problem then repeat initial calibration.
Laboratory Control Sample (LCS)	Once per initial calibration, and with each analytical batch (maximum of 20 samples).	See Table 6-1.2.	Investigate the problem and if warranted, analyze a new analytical curve for the out-of-limits compound. (except for compounds noted in Table 6-1.2.)

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV)	At the start of every shift immediately after the BFB tune check.	<b>For SPCCs:</b> see "CCV criteria" column <b>For CCCs:</b> %D ≤ 25% for VOST tubes and ≤ 20% for condensates.	Investigate and correct the problem, up to and including recalibration if necessary.
Internal Standards (IS)	As each standard, blank, and sample is being loaded.	<b>For CCVs:</b> area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL. <b>For blanks, samples and non-CCV QC Checks:</b> area counts 60 - 140%, RT w/in 20 sec. of RT in CCV.	<b>CCV:</b> inspect and correct system prior to sample analysis. <b>For blanks:</b> inspect the system and re-analyze the blank. <b>For condensates:</b> re-analyze; if out again, flag data. <b>For VOST:</b> flag the data, evaluate system and correct problem before proceeding.
Surrogates	With all samples and QC.	See Table 6-1.5.	Same as for Internal Standards.
Laboratory Blanks	Immediately after the calibration standard or after samples with high concentrations (≥ 5000 ng).	Results less than laboratory reporting limit	Inspect the system and re-analyze the blank.
(MS/MSD)	Once/batch of condensate samples.	See Table 6-1.3.	Q-flag and narrate.

**6.2 TO-14A AND TO-3 - BTEX AND TPH**

This method involves GC analysis of whole air samples collected in Summa™ canisters or Tedlar bags. Samples are analyzed for Benzene, Toluene, Ethylbenzene, Xylenes, and Total Petroleum Hydrocarbons (TPH) using EPA Method TO-14A or EPA Method TO-3 protocols. Samples are analyzed using a Photo Ionization Detector (PID) and a Flame Ionization Detector (FID). Depending on the client's request, TPH is analyzed and referenced to either gasoline or jet fuel.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client

proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version for each of these methods. The method modifications, standard target analyte list, RL, QC criteria, and QC summary can be found in the following tables.

**Table 6.2.1 Summary of Method Modifications**

<b>Requirement</b>	<b>EPA Method TO-14A</b>	<b>Air Toxics Ltd. Modifications</b>
Sample Drying System*	Nafion Dryer	Multi-bed sorbent
Sample collection containers	Specially treated stainless steel canisters.	Method TO-14A is validated for samples collected in specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of the method and not recommended for ambient or indoor air samples. Associated results are considered qualified.

**Table 6.2.2 Summary of Method Modifications**

<b>Requirement</b>	<b>EPA Method TO-3</b>	<b>Air Toxics Ltd. Modifications</b>
Sample Collection	In-line field method	Collection of sample in specially treated canisters or alternative containers for transport to and analysis by an off-site laboratory
Preparation of Standards	Levels achieved through dilution of gas mixture.	Levels achieved through loading various volumes of the gas mixture.
Initial Calibration Calculation	4-point calibration using a linear regression model.	5-point calibration using average Response Factor.
Initial Calibration Frequency	Weekly.	When daily calibration standard recovery is outside 75-125%, or upon significant changes to the procedure or instrumentation.

Requirement	EPA Method TO-3	Air Toxics Ltd. Modifications
Daily Calibration Standard Frequency	Prior to sample analysis and every 4-6 hrs.	Prior to sample analysis and after the analytical batch $\leq$ 20 samples.
Minimum Detection Limit (MDL)	Calculated using the equation $DL = A + 3.3S$ , where A is intercept of calibration line and S is the standard deviation of at least 3 reps of low level standard.	40 CFR Pt. 136 App. B.
Sample preconcentration and moisture management	Cryogenic preconcentrator with a Nafion dryer	Multi-bed Sorbent system

**Table 6.2.3 Method TO-14A/TO-3 Standard Analyte List**

Analyte	RL (ppmv)	Acceptance Criteria		
		ICAL (%RSD)	LCS & CCV (%R)	Precision (%RPD)
Benzene	0.001	$\leq 30$	$\pm 25$	$\leq 25$
Toluene	0.001	$\leq 30$	$\pm 25$	$\leq 25$
Ethyl Benzene	0.001	$\leq 30$	$\pm 25$	$\leq 25$
Total Xylenes	0.001	$\leq 30$	$\pm 25$	$\leq 25$
TPH* (Gasoline Range)	0.025	$\leq 30$	$\pm 25$	$\leq 25$
TPH** (JP 4 Range)	0.025	$\leq 30$	$\pm 25$	$\leq 25$

\* TPH referenced to Gasoline (MW = 100)

\*\* TPH referenced to JP 4 (MW = 156)

**Table 6.2.4 Surrogate**

Surrogate	PID Accuracy (%R)	FID Accuracy (%R)
Fluorobenzene	75-125%	75-150%

**Table 6-2.5. Summary of Calibration and QC Procedures for Method TO-14A/TO-3 (BTEX & TPH)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five Point Calibration (ICAL)	Prior to sample Analysis.	%RSD $\leq 30$ .	Repeat the calibration.
Laboratory Control Sample (LCS)	With each initial calibration, and with each analytical batch.	$\pm 25\%$ of the expected value.	Check the system and re-analyze the standard. Re-prepare the standard or re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis.	$\pm 25\%$ of the expected value.	For initial CCV: Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Mid/End Check	At the end of the analytical batch, not to exceed 20 samples.	±25% of the expected value.	Check system and re-analyze the standard. If the 2nd analysis fails, identify and correct the problem, then re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples.	Results less than the laboratory Reporting Limit.	Repeat the Laboratory Blank. If the re-analysis of the Lab Blank contains compounds above but at less than 5 X the reporting limit, sample analysis may proceed and the associated sample results will be reported with a B flag.
Surrogate Spikes	As each standard, blank, and sample is being loaded.	75-125% recovery on the PID, 75-150% on the FID.	Low surrogate recovery results in re-analysis (at a higher dilution if high levels of moisture are present). If recovery is out and still low, report the analysis with the better recovery and flag. Because of TPH interference, high surrogate recoveries do not result in re-analysis. Data is flagged to note high recovery.
Laboratory Duplicates	10% of the samples.	RPD ≤ 25% for detections >5 X's RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate the data.

**6.3 TO-4A/TO-10A – PESTICIDES AND PCBs**

This method involves GC analysis of Pesticides and Aroclor Polychlorinated Biphenyls (PCBs) in ambient air samples collected on polyurethane foam (PUF) cartridges.

Adsorbent PUF cartridges are cleaned using solvents and vacuum dried. Cartridges are sent to the field wrapped tightly in aluminum foil to prevent degradation by ultraviolet (UV) light. The PUF cartridges are batch certified for cleanliness prior to shipping. In addition, the laboratory analyzes one clean PUF cartridge for each extraction set to serve as a Laboratory Blank.

A high volume sampler is used for TO-4A and a low volume sampler is used for method TO-10A.

The filters and cartridges are prepared for analysis by either Soxhlet or Pressurized Fluid Extraction (PFE) by EPA Method 3545A. The extract is concentrated, exchanged into Hexane and concentrated again to final

volume. Analysis is performed using a GC/ECD (Electron Capture Detector).

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. For the extraction process, the non-standard compound recovery is evaluated in the extracted laboratory control spike. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.3.1 Summary of Method Modifications for TO-4A/TO-10A**

<i>Requirement</i>	<b>EPA Method TO-4A/TO-10A</b>	<b>Air Toxics Ltd. Modifications</b>
Extraction Solvent	10 % (5 % TO-10A) Diethyl Ether in Hexane.	DCM, exchanging to Hexane during the concentration step.
Reagent Blank	Set up extraction system without filter/PUF; reflux with solvent.	No Reagent Blank is extracted. Reagent lots are certified as acceptable prior to use.
Media certification (TO-10A only)	< 0.01 µg for single peak analytes, < 0.1 µg for PCBs.	< Reporting Limit for all analytes.
Frequency of Continuing Calibration Verification	Every 10 samples.	Every 20 samples with internal standard.
PCB Quantitation	Requires a minimum of 5 peaks.	Use 4 peaks for quantitation.

Table 6.3.2 Methods TO-4A/TO-10A Pesticides and PCBs Reporting and QC Limits

Analyte	RL (µg)	Acceptance Criteria				
		ICAL (%RSD)	ISCV (%R)	CCV (%D)	LCS <sup>②</sup> (%R)	Precision (%RPD)
4,4'-DDD	0.10	< 20	± 15	± 15		≤ 25%
4,4'-DDE	0.10	< 20	± 15	± 15		≤ 25%
4,4'-DDT	0.10	< 20	± 15	± 15	65-125	≤ 25%
4,4'-Methoxychlor	1.0	< 20	± 15	± 15		≤ 25%
Aldrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
alpha-BHC	0.10	< 20	± 15	± 15		≤ 25%
alpha-Chlordane	0.10	< 20	± 15	± 15		≤ 25%
Aroclor 1016/1242	1.0	< 20	± 15	± 15	65-125	≤ 25%
Aroclor 1221 <sup>①</sup>	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1232 <sup>①</sup>	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1248 <sup>①</sup>	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1254 <sup>①</sup>	1.0	< 20	± 15	± 15		≤ 25%
Aroclor 1260	1.0	< 20	± 15	± 15	65-125	≤ 25%
beta-BHC	0.10	< 20	± 15	± 15		≤ 25%
delta-BHC	0.10	< 20	± 15	± 15		≤ 25%
Dieldrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
Endosulfan I	0.10	< 20	± 15	± 15		≤ 25%
Endosulfan II	0.10	< 20	± 15	± 15		≤ 25%
Endosulfan Sulfate	0.10	< 20	± 15	± 15		≤ 25%
Endrin	0.10	< 20	± 15	± 15	65-125	≤ 25%
Endrin Aldehyde	0.10	< 20	± 15	± 15		≤ 25%
Endrin Ketone	0.10	< 20	± 15	± 15		≤ 25%
gamma-BHC (Lindane)	0.10	< 20	± 15	± 15	65-125	≤ 25%
gamma-Chlordane	0.10	< 20	± 15	± 15		≤ 25%
Heptachlor	0.10	< 20	± 15	± 15	65-125	≤ 25%
Heptachlor Epoxide	0.10	< 20	± 15	± 15		≤ 25%
Technical Chlordane <sup>④</sup>	1.0	< 20	± 15	± 15		≤ 25%
Toxaphene <sup>①</sup>	1.0	< 20	± 15	± 15		≤ 25%

① The noted multi-component compounds use a one-point calibration.

② Recovery limits are derived from Compendium Method TO-10A January, 1999.

③ Recovery limits are for extracted samples only. Non-extracted samples use limits of 85 – 115 %R.

④ Not routinely reported, available at client request

Table 6.3.3 Surrogates<sup>③</sup>

Surrogate	%R
TCMX	60 – 120 <sup>②</sup>
DCB	60 – 120 <sup>②</sup>

**Table 6.3.4 Summary of Calibration and QC Procedures for Methods TO-4A/TO-10A**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Five Point ICAL*	Prior to sample analysis.	%RSD $\leq$ 20 for each compound or average %RSD $\leq$ 20.	Use linear regression per SW-846 or re-calibrate.
Independent Source Calibration Verification (ISCV)	After each Initial Calibration.	Recovery of an individual component or the average of all the target components for a list of 5 or more target components within 85 to 115 % recovery. Not to exceed 75-125% for any individual compounds.	Investigate the source of discrepancy, including re-preparation and re-analysis of standard. Re-calibrate if needed.
Breakdown Check	Daily, CCV Pesticides only.	Degradation $\leq$ 15%.	Perform maintenance. Repeat breakdown check.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, every 20 samples, and at the end of the analysis sequence.	Recovery of an individual component or the average of all the Pesticide target components for a list of 5 or more target components, within 15% of the expected values. Not to exceed 75-125% for any individual compounds.	Analyze new ICAL and/or prepare fresh standards. If the standard analyzed is high and associated samples are ND, "Q" flag the high recoveries. If the standard analyzed is low, re-analyze all samples.
Laboratory Control Spike (LCS)	Extracted with each set of up to 20 samples.	As mentioned in Table 6-3.2.	Analyze another aliquot. If it still fails, "Q" flag the compounds outside the control limits.
Surrogates	All samples, QC, and blanks prior to extraction.	As mentioned in Table 6-3.3	Analyze another aliquot, if it still fails, "Q" flag the compounds outside the control limits.
Internal Standard	With all analyses.	CCV 50-200% compared to midpoint of ICAL; Samples 50-200% compared to first CCV of the daily analytical batch.	Analyze another 100 $\mu$ L aliquot. If a CCV fails, correct problem before proceeding. If a sample fails, analyze a second time. If it still fails, dilute the sample until IS meet the criteria. Narrate the matrix interference.
Laboratory Blanks	With each set of up to 20 samples extracted.	Results less than the Laboratory reporting limit.	Analyze another aliquot. If it still fails, "B" flag the compounds that do not meet the acceptance criteria.
Laboratory Duplicates	10% of the samples.	RPD $\leq$ 25% for detections $>$ 5 X's RL.	Analyze sample a 3 <sup>rd</sup> time. If criteria are still not met, narrate the data.
Second-Column Confirmation	100% for all positive results, for both Pesticide/PCB analyses.	Same as for initial or primary column analysis.	Same as for initial or primary column analysis.

\* A single point calibration is performed for Technical Chlordane, Toxaphene, and certain Aroclors.

**6.4 TO-5, TO-11A, METHOD 0011,  
 CARB 430 – ALDEHYDES,  
 AND KETONES**

These methods involve High-Pressure Liquid Chromatography (HPLC) analysis of Aldehydes and Ketones in stationary and ambient air samples. The sampling media consist either of various-sized impingers containing 2,4-Dinitrophenylhydrazine (DNPH) reagent, or a Sep-PAK (silica) cartridge coated in-situ with a solution of DNPH. Aldehydes and Ketones are readily converted to a stable Hydrazone derivative. The impinger contents are extracted with a 70:30 Methylene Chloride/Hexane solution or Methylene Chloride only, concentrated, solvent exchanged and analyzed on the HPLC. The Sep-PAK cartridges are eluted with Acetonitrile using gravity feed technique. Analysis is performed by reverse phase HPLC with UV detection at 360 nm.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. For the extraction process, the non-standard compound recovery is evaluated in the extracted laboratory control spike. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.4.1 Summary of TO-5/CARB 430 Method Modifications**

<b>Requirement</b>	<b>TO-5/CARB 430</b>	<b>Air Toxics Ltd. Modifications</b>
Initial Calibration (ICAL)	<b>TO-5:</b> Linear regression, $R \geq 0.999$ .	Average Response Factor (RF), composite % RSD $\leq 10$ . Linear regression is performed when requested.
Sample Quantitation	Use daily RF.	Use ICAL RF.
Calibration Standard Precision	% RSD $\pm 10\%$ .	Recovery of all Continuing Cal. standards must be 90% - 110%.
Retention Time (RT) Precision	%RPD $< 2\%$ for daily calibration standards.	RT windows determined by bracketing standards.
Limit of Detection	<b>CARB 430:</b> The "limit of detection" is defined as the upper bound of the 95% confidence interval for the analysis of at least 4 reagent blanks.	Detection Limit is based on the current MDL study which is calculated from a minimum of 7 extracted spikes following 40 CFR Part 136 Appendix B.
Field Blank Subtraction	<b>CARB 430:</b> Subtract the average of the field blanks from the samples.	The samples and Field Blanks are not blank subtracted.

Requirement	TO-5/CARB 430	Air Toxics Ltd. Modifications
Laboratory Control Spike (LCS)	<b>CARB 430:</b> If the LCS is out it must be re-extracted until it is in or recalibrate.	The LCS is only extracted once with out of control recoveries flagged.
UV Absorption Detector	<b>TO-5:</b> Operate at 370 nm.	Operate at 360 nm.
Mobile Phase	<b>TO-5:</b> Methanol/Water.	Acetonitrile/Water.

**Table 6.4.2 Summary of Method TO-11A Modifications**

Requirement	TO-11A	<i>Air Toxics Ltd. Modifications</i>
Initial Calibration Curve (ICAL)	Multi-point using linear regression performed every 6 months.	Multi-point using average Response Factor; re-calibration if daily cal. fails, major maintenance or column change. Linear regression is performed when requested.
ICAL Criteria	R2 for curve > 0.999	%RSD ≤ 10% unless Linear regression is requested.
Blank Subtraction	Average blank concentrations calculated. Blank value subtracted from sample result.	One Lab Blank is analyzed per batch; no blank subtraction performed on samples.

**Table 6.4.3 Methods TO-5/CARB 430, TO-11A, and Method 0011 Standard Analyte List**

Analyte	TO-5/ Method 0011/ CARB430 RL (µg)	TO-11A RL (µg)	Acceptance Criteria		
			ICAL (%RSD)	ISCV (%R)	CCV (%R)
Formaldehyde	0.5	0.05	≤ 10	± 15	± 10
Acetaldehyde	0.5	0.10	≤ 10	± 15	± 10
Acrolein <sup>a</sup>	0.5	N/A	≤ 10	± 15	± 10
Acetone*	NA	0.25	≤ 10	± 15	± 10
Propanal	0.5	0.25	≤ 10	± 15	± 10
Crotonaldehyde*	0.5	0.25	≤ 10	± 15	± 10
n- Butyraldehyde <sup>b</sup>	0.5	0.25	≤ 10	± 15	± 10
Isopentanal*	0.5	0.25	≤ 10	± 15	± 10
Pentanal	0.5	0.25	≤ 10	± 15	± 10
m,p-Tolualdehyde*	0.5	0.25	≤ 10	± 15	± 10
o-Tolualdehyde*	0.5	0.25	≤ 10	± 15	± 10
Hexanal	0.5	0.25	≤ 10	± 15	± 10
Dimethylbenzaldehyde **	0.5	0.25	≤ 10	± 15	± 10

- <sup>a</sup> *Because its derivative is not stable, when the target analyte list includes Acrolein, the sample will need to be extracted in field. A special order should be placed with the laboratory during the project set up stage.*
- <sup>b</sup> *Methyl Ethyl Ketone, Iso-Butyraldehyde and the n-Butyraldehydes co-elute(report as n-Butyraldehyde)*
- <sup>c</sup> *Not recommended for TO-11A*
- <sup>\*</sup> *Not included in the extracted LCS compound list for methods CARB 430/TO-5 and method 0011.*
- <sup>\*\*</sup> *Special request compound*

**Table 6.4.4 Summary of Calibration and QC Procedures for Methods TO-5/CARB 430, TO-11A, and Method 0011**

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Five Point Initial Calibration Curve (ICAL)	Analyzed in triplicate prior to sample analysis	%RSD ≤ 10.	Repeat calibration.
Instrument LCS	With each ICAL	%R = 85-115%.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis, after a maximum of every 10 injections, and at the end of the analytical batch.	Within ± 10% of the expected value.	Check the system and re-analyze the standard. If the criteria cannot be met, re-calibrate the instrument. If the standard is biased low, re-analyze all samples since last acceptable CCV. If biased high and samples are “ND”, re-analysis is not required. Q-flag high recoveries.
Instrument (Solvent) Blank Analysis	Following analysis of Standards.	Results less than the laboratory RL.	Inspect the system and Re-analyze the blank.
Laboratory Duplicates	10% of samples.	RPD ≤ 25% for detections >5 Xs RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

## 6.5 TO-12-NMOC

This method involves GC analysis of whole air samples collected in Summa™ canisters or Tedlar bags. Samples are analyzed for Non-Methane Organic Compounds (NMOC) using EPA Method TO-12 protocols. After concentration on a sorbent bed, samples are analyzed using a Flame Ionization Detector (FID). This method is used when speciation is not required.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds

with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Reporting Limits, QC criteria, and QC summary can be found in the following tables:

**Table 6.5.1 Summary of Method Modifications**

Requirement	EPA Method TO-12	Air Toxics Ltd. Modifications
Reporting Limit	0.02 ppmc.	0.010 ppmv.
Reported Units	Ppmc.	Ppmv, or if ref to CH <sub>4</sub> , multiply by 7. Units in ppmvc.
Initial Calibration	Five levels – each level three runs with %RSD < 3%; Linearity criterion not specified.	Minimum of three single levels; %RSD ≤ 30%.
Sample Analysis Frequency	Duplicate analysis with RPD < 5%, report average result .	Single analysis. Duplicate 10% of samples with RPD ≤ 25% for detections > 5 X's the RL.
Sample Hold Time	None specified.	Canister 30 days, Tedlar bags 3 days.
Column	GC column not used.	GC column used for analysis.

**Table 6.5.2 Method TO – 12 Standard Analyte List**

Analyte	RL (ppmv)	ICAL (%RSD)	CCV %D	LCS %R	Duplicates %RPD
TNMOC ref. to Heptane	0.010	≤ 30	± 25	75-125	≤ 25

Heptane MW = 100

**Table 6.5.3 Summary of Calibration and QC Procedures for Method TO-12**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to sample analysis.	% RSD $\leq$ 30.	Repeat the calibration.
Laboratory Control Sample (LCS)	With each initial calibration and analytical batch.	75-125% of the expected value.	Check the system and re-analyze the Standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples or at the end of the analytical sequence.	% Difference $\pm$ 25.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met. Re-analyze all samples since the last acceptable CCV.
Laboratory Blank	In between analysis of standards and project samples.	Results less than laboratory reporting limit.	Repeat the Laboratory Blank. If the re-analysis of the Lab Blank contains compounds above but at less than 5 X the reporting limit, sample analysis may proceed and the associated sample results will be reported with a B flag.
Sample Duplicates	10% of the samples.	$\leq$ 25% for detections > 5X's the RL.	Re-analyze the sample for a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

**6.6 TO-13A AND 8270C – SEMIVOLATILE COMPOUNDS**

This method involves GC/MS full scan or SIM mode analysis of semi-volatile organic compounds in ambient air samples collected on PUF/XAD2 cartridges. In relation to the prescribed media, sampling and collection efficiency for compounds not listed in TO-13A has not been evaluated. Samples are analyzed for Polynuclear Aromatic Hydrocarbons (PAHs) using a quadrupole GC/MS in full scan or SIM mode by TO-13A protocol. In addition, the target compound list is often extended to include analysis of Method 8270 semi-volatile compounds. Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

Certain compounds are not included in ATL’s standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been

made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Certain compounds are not included in ATL’s standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. For the extraction process, the non-standard compound recovery is evaluated in the extracted laboratory control spike. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. also performs semi-volatile analysis by SW-846 Method 8270C. The extraction process of MM5 trains follows SW846 Method 3542, and the QC criteria differ from Method TO-13A analysis. The QC criteria and QC summary tables for Method 8270C analysis are in the section following the TO-13A tables.

**Table 6.6.1 Summary of Method Modifications for TO-13A**

<b>Requirements</b>	<b>EPA Method TO-13A</b>	<b>Air Toxics Ltd. Modifications</b>
Extraction Solvent	10% ether in hexane for PUF; DCM for XAD sorbent. Final extract in hexane.	DCM for PUF/XAD cartridge and XAD sorbent. Final extract in DCM.
Glassware Cleaning	Muffle furnace is utilized.	Solvent cleaning procedure is used.
Extraction Technique	Soxhlet extraction.	Soxhlet extraction or pressurized fluid extraction (PFE).
Reporting List	19 PAHs.	See Tables 6-7.2 & 6-7.3.
Calibration range:	0.1-2.5 µg/mL in Hexane	1.0-160 µg/mL in Methylene chloride for quad or 0.1-40 µg/mL for SIM.

Requirements	EPA Method TO-13A	Air Toxics Ltd. Modifications
Surrogate	Field surrogates: Fluoranthene-d10 and Benzo(a)pyrene-d12.	Field surrogates: provided upon request.
Solvent Process Blank	One each analytical batch.	Not performed: each solvent lot is certified.
Method Blank	< MDL.	<Reporting Limit.

**Table 6.6.2 Modified Method TO-13A**

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
2-Chloronaphthalene*	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
2-Methylnaphthalene*	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
Acenaphthylene	0.1	1.0	1.3	≤ 30	± 30	± 30	≤ 25%
Acenaphthene	0.1	1.0	0.8	≤ 30	± 30	± 30	≤ 25%
Anthracene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(a)anthracene	0.1	1.0	0.8	≤ 30	± 30	± 30	≤ 25%
Benzo(e)pyrene*	0.1	1.0	NA	≤ 30	± 30	± 30	≤ 25%
Benzo(a)pyrene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(b)fluoranthene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Benzo(g,h,i)perylene	0.1	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Benzo(k)fluoranthene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Chrysene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Dibenz(a,h)anthracene	0.1	1.0	0.4	≤ 30	± 30	± 30	≤ 25%
Fluoranthene	0.1	1.0	0.6	≤ 30	± 30	± 30	≤ 25%
Fluorene	0.1	1.0	0.9	≤ 30	± 30	± 30	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.1	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Naphthalene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Phenanthrene	0.1	1.0	0.7	≤ 30	± 30	± 30	≤ 25%
Pyrene	0.1	1.0	0.6	≤ 30	± 30	± 30	≤ 25%

\* Not included in the TO-13A method.

The following two compounds can be analyzed upon client's request.

Analyte	SIM RL (µg)	RL (µg)	Minimum ICAL RRF	ICAL (%RSD)	ISCV (%R)	CCV (%R)	Precision (%RPD)
Perylene	NA	1.0	0.5	≤ 30	± 30	± 30	≤ 25%
Coronene	NA	1.0	0.7	≤ 30	± 30	± 30	≤ 25%

**Table 6.6.3 Modified Method TO-13A-Extended**

Analyte	Minimum ICAL RRF	RL (µg)	ICAL <sup>(1)</sup> (%RSD)	ISCV (%R)	Precision %RPD
1,2,4-Trichlorobenzene	NA	1.0	< 30	± 30	< 25%
1,2-Dichlorobenzene	NA	1.0	< 30	± 30	< 25%
1,3-Dichlorobenzene	NA	1.0	< 30	± 30	< 25%
1,4-Dichlorobenzene - CCC	NA	1.0	< 30	± 30	< 25%
2,4,5-Trichlorophenol	NA	5.0	< 30	± 30	< 25%
2,4,6-Trichlorophenol - CCC	NA	5.0	< 30	± 30	< 25%
2,4-Dichlorophenol - CCC	NA	5.0	< 30	± 30	< 25%
2,4-Dimethylphenol	NA	5.0	< 30	± 30	< 25%
2,4-Dinitrophenol - SPCC	0.05	20	< 30	± 30	< 25%
2,4-Dinitrotoluene	NA	5.0	< 30	± 30	< 25%
2,6-Dinitrotoluene	NA	5.0	< 30	± 30	< 25%
2-Chloronaphthalene	NA	1.0	< 30	± 30	< 25%
2-Chlorophenol	NA	5.0	< 30	± 30	< 25%
2-Methylnaphthalene	NA	1.0	< 30	± 30	< 25%
2-Methylphenol	NA	5.0	< 30	± 30	< 25%
2-Nitroaniline	NA	10	< 30	± 30	< 25%
2-Nitrophenol – CCC	NA	5.0	< 30	± 30	< 25%
3,3-Dichlorobenzidine	NA	20	< 30	± 30	< 25%
3-Nitroaniline	NA	10	< 30	± 30	< 25%
4,6-Dinitro-2-methylphenol	NA	10	< 30	± 30	< 25%
4-Bromophenyl-phenyl ether	NA	1.0	< 30	± 30	< 25%
4-Chloro-3-methylphenol - CCC	NA	5.0	< 30	± 30	< 25%
4-Chloroaniline	NA	10	< 30	± 30	< 25%
4-Chlorophenyl-phenyl ether	NA	1.0	< 30	± 30	< 25%
4-Methylphenol	NA	5.0	< 30	± 30	< 25%
4-Nitroaniline	NA	10	< 30	± 30	< 25%
4-Nitrophenol – SPCC	0.05	20	< 30	± 30	< 25%
Acenaphthylene	1.3	1.0	< 30	± 30	< 25%
Acenaphthene – CCC	0.8	1.0	< 30	± 30	< 25%
Anthracene	0.7	1.0	< 30	± 30	< 25%
Benzo(a)anthracene	NA	1.0	< 30	± 30	< 25%
Benzo(a)pyrene - CCC	0.7	1.0	< 30	± 30	< 25%
Benzo(e)pyrene	0.5	1.0	< 30	± 30	< 25%
Benzo(b)fluoranthene	0.7	1.0	< 30	± 30	< 25%
Benzo(g,h,i)perylene	NA	1.0	< 30	± 30	< 25%
Benzo(k)fluoranthene	NA	1.0	< 30	± 30	< 25%
Benzoic Acid	NA	30	< 30	± 30	< 25%
Bis(2-Chloroethoxy) Methane	NA	1.0	< 30	± 30	< 25%
Bis(2-Chloroisopropyl) Ether	NA	1.0	< 30	± 30	< 25%
Bis(2-Chloroethyl) Ether	NA	1.0	< 30	± 30	< 25%
Bis(2-Ethylhexyl)phthalate	NA	5.0	< 30	± 30	< 25%
Butylbenzylphthalate	NA	5.0	< 30	± 30	< 25%

Analyte	Minimum ICAL RRF	RL (µg)	ICAL <sup>(1)</sup> (%RSD)	ISCV (%R)	Precision %RPD
Chrysene	0.7	1.0	≤ 30	± 30	≤ 25%
di-n-Butylphthalate	NA	5.0	≤ 30	± 30	≤ 25%
di-n-Octylphthalate - CCC	NA	5.0	≤ 30	± 30	≤ 25%
Dibenz(a,h)anthracene	0.4	1.0	≤ 30	± 30	≤ 25%
Dibenzofuran	NA	1.0	≤ 30	± 30	≤ 25%
Diethylphthalate	NA	5.0	≤ 30	± 30	≤ 25%
Dimethylphthalate	NA	5.0	≤ 30	± 30	≤ 25%
Fluoranthene – CCC	0.6	1.0	≤ 30	± 30	≤ 25%
Fluorene	0.9	1.0	≤ 30	± 30	≤ 25%
Hexachlorobenzene	NA	1.0	≤ 30	± 30	≤ 25%
Hexachlorobutadiene - CCC	NA	1.0	≤ 30	± 30	≤ 25%
Hexachlorocyclopentadiene-SPCC	0.05	20	≤ 30	± 30	≤ 25%
Hexachloroethane	NA	1.0	≤ 30	± 30	≤ 25%
Indeno(1,2,3-c,d)pyrene	0.5	1.0	≤ 30	± 30	≤ 25%
Isophorone	NA	1.0	≤ 30	± 30	≤ 25%
n-Nitroso-di-n-propylamine-SPCC	0.05	1.0	≤ 30	± 30	≤ 25%
n-Nitrosodiphenylamine – CCC	NA	10	≤ 30	± 30	≤ 25%
Naphthalene	0.7	1.0	≤ 30	± 30	≤ 25%
Nitrobenzene	NA	1.0	≤ 30	± 30	≤ 25%
Pentachlorophenol – CCC	NA	20	≤ 30	± 30	≤ 25%
Phenanthrene	0.7	1.0	≤ 30	± 30	≤ 25%
Phenol – CCC	NA	5.0	≤ 30	± 30	≤ 25%
Pyrene	0.6	1.0	≤ 30	± 30	≤ 25%

<sup>(1)</sup> With 10% exception not to exceed 40%

**Table 6.6.4 Surrogates (Full Scan)**

Analyte	(%R)
2,4,6-Tribromophenol	50 – 150
2-Fluorophenol	50 – 150
Nitrobenzene-d <sub>5</sub>	50 – 150
Phenol-d <sub>5</sub>	50 – 150
Fluorene-d <sub>10</sub>	60 – 120
Pyrene-d <sub>10</sub>	60 – 120

**Table 6.6.5 Internal Standards**

Analyte	(%)
Acenaphthene-d <sub>10</sub>	50 – 200
Chrysene-d <sub>12</sub>	50 – 200
1,4-Dichlorobenzene-d <sub>4</sub>	50 – 200
Naphthalene-d <sub>8</sub>	50 – 200
Perylene-d <sub>12</sub>	50 – 200
Phenanthrene-d <sub>10</sub>	50 – 200

**Table 6.6.6. TO-13A-Surrogates (Standard and SIM)**

Analyte	Accuracy (% R)*
Fluorene-d <sub>10</sub>	60 - 120
Pyrene-d <sub>10</sub>	60 - 120

**Table 6.6.7 Extracted Laboratory Control Spikes for Modified TO-13A-Extended**

Analyte	(%R)
1,2,4-Trichlorobenzene***	50 – 150
1,4-Dichlorobenzene***	50 – 150
2,4-Dinitrotoluene***	50 – 150
2-Chlorophenol***	50 – 150
4-Chloro-3-methylphenol***	50 – 150
4-Nitrophenol***	50 – 150
Acenaphthene*	60 – 120
N-Nitroso-di-n-propylamine***	50 – 150
Analyte	(%R)
Pentachlorophenol**	22 – 109
Phenol***	50 – 150
Pyrene*	60 – 120

\* The LCS and Surrogate limits are derived from Compendium Method TO-13A Sections 13.3.7.4 and 13.4.6.3 January, 1999. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 – 150 % are applied.

\*\* Pentachlorophenol is not included in Compendium Method TO-13A and has been shown to be erratically recovered from XAD media therefore historical Control Limits are used. Limits are updated periodically as needed.

\*\*\* Compounds outside of the TO-13A method

**Table 6.6.8 Extracted Laboratory Control Samples for TO-13A (PAHs) in Full Scan and SIM**

Analyte	(%R)
Napthalene	60 – 120
Acenaphthylene	60 – 120
Acenaphthene	60 – 120
Flourene	60 – 120
Phenanthrene	60 – 120
Anthracene	60 – 120
Fluoranthene	60 – 120
Pyrene	60 – 120
Benzo (a) anthracene	60 – 120
Chrysene	60 – 120
Benzo (b) flouranthene	60 – 120
Benzo (k) flouranthene	60 – 120
Benzo (a) pyrene	60 – 120
Indeno (1,2,3-cd) pyrene	60 – 120
Dibenzo (a,h) anthracene	60 – 120
Benoz (g,,h,i) perylene	60 – 120

**Table 6.6.9 Summary of Calibration and QC Procedures for EPA Method TO-13A**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hrs.	SW-846 tuning criteria for semivolatiles analysis. DDT% Breakdown < 20%	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	ICAL criteria in tables 6-7.2 and 6-7.3	Correct problem then repeat initial calibration.
ICAL LCS	All analytes – Once per initial calibration.	All target compound recoveries must be between 70 – 130%.	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock immediately after the DFTPP tune check.	PAHs list: meet min. RRF requirement PAHs list/ short list %D ≤ 30% Semivol full list: SPCCs: RF ≥ 0.050 %D ≤ 30% with 10% exception not to exceed 40%. Flag all results outside of compliance with the exception of high bias associated with non-detects.	Investigate and correct the problem, up to and including re-calibration if necessary. High bias associated with non-detects in samples will not result in re-analysis.
Internal Standards (IS)	As each standard, blank, and sample is being aliquoted.	For CCV: Area count within 50 to 200% of the mid point of ICAL.  For blanks, samples and non-CCV QC Checks: retention times within ± 0.33 minutes (20 seconds) and area counts within 50 to 200% of the CCV.	For CCVs: Investigate and correct the problem before proceeding with sample analysis. If interferences are present, a secondary ion may be selected. <u>For blanks:</u> inspect the system and re-analyze the blank. <u>For samples and non-CCV QC:</u> unless there is obvious matrix effect, re-analyze the samples and dilute the sample until the IS meet the criteria, narrate the data to indicate interference.
Surrogates	With all samples and blanks prior to extraction.	See Table 6-7.4.	A new aliquot of the extract is analyzed. If Surrogate recoveries are out-of-control a second time, data is flagged and narrated. Re-analysis is not necessary for obvious matrix effects (data is flagged for out-of-control surrogate recoveries). Air samples cannot be re-extracted.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Extracted LCS	With each set of up to 20 extracted samples.	See LCS Criteria in tables 6-7.7 and 6-7.8.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.
Laboratory Blank	With each set of up to 20 extracted samples.	Results less than laboratory reporting limit.	Flag the data.
Solvent Blanks	When samples that are extracted together are analyzed on different analytical shifts.	All target compounds below the reporting limit.	Flag the data.
Laboratory Duplicates	10% of the samples.	RPD $\leq$ 25% for all hits > 5X RLs.	Narrate the data.

**Table 6.6.10 Summary of Method Modifications for EPA Methods 3510/3542 and 8270C**

Requirements	EPA Method 8270C	Air Toxics Ltd. Modifications
Linearity of ICAL	Use mean RF for non-CCC compounds if %RSD $\leq$ 15%. If %RSD > 15%, use a) linear regression equation that does not pass through the origin. R $\geq$ 0.99, or b) non-linear (i.e., 6 points for a quadratic model).	Use mean RF for non-CCC compounds when %RSD $\leq$ 30%.
RT for CCV	Within +/- 30 seconds of the mid-point standard from the initial curve.	Frequent column maintenance results in RT shift; therefore this requirement is not practical.

**Table 6.6.11 SW-846 Modified Method 8270C Standard Analyte List**

Analyte	RL ( $\mu$ g)	Acceptance Criteria			
		ICAL (%RSD) <sup>①</sup>	ISCV (%R) <sup>②</sup>	CCV <sup>③</sup>	Precision\%RPF
1,2,4-Trichlorobenzene	1.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
1,2-Dichlorobenzene	1.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
1,3-Dichlorobenzene	1.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
1,4-Dichlorobenzene - CCC	1.0	$\leq$ 30	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,4,5-Trichlorophenol	5.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,4,6-Trichlorophenol - CCC	5.0	$\leq$ 30	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,4-Dichlorophenol - CCC	5.0	$\leq$ 30	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,4-Dimethylphenol	5.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,4-Dinitrophenol - SPCC	20	$\leq$ 15	$\pm$ 30	<b>RF &gt; 0.050</b>	$\leq$ 25%
2,4-Dinitrotoluene	5.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2,6-Dinitrotoluene	5.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%
2-Chloronaphthalene	1.0	$\leq$ 15	$\pm$ 30	%D $\leq$ 20%	$\leq$ 25%

Analyte	RL (µg)	Acceptance Criteria			
		ICAL (%RSD) <sup>①</sup>	ISCV (%R) <sup>②</sup>	CCV <sup>③</sup>	Precision\%RP F
2-Chlorophenol	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
2-Methylnaphthalene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
2-Methylphenol	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
2-Nitroaniline	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
2-Nitrophenol – CCC	5.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
3,3-Dichlorobenzidine	20	≤ 15	+ 30	%D ≤ 20%	≤ 25%
3-Nitroaniline	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
4,6-Dinitro-2-methylphenol	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
4-Bromophenyl-phenyl ether	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
4-Chloro-3-methylphenol - CCC	5.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
4-Chloroaniline	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
4-Chlorophenyl-phenyl ether	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
4-Methylphenol	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
4-Nitroaniline	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
4-Nitrophenol – SPCC	20	≤ 15	+ 30	<b>RF &gt; 0.050</b>	≤ 25%
Acenaphthylene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Acenaphthene – CCC	1.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Anthracene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Benzo(a)anthracene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Benzo(a)pyrene - CCC	1.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Benzo(b)fluoranthene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Benzo(g,h,i)perylene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Benzo(k)fluoranthene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Benzoic Acid	30	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Bis(2-Chloroethoxy) Methane	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Bis(2-Chloroisopropyl) Ether	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Bis(2-Chloroethyl) Ether	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Bis(2-Ethylhexyl)phthalate	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Butylbenzylphthalate	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Chrysene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
di-n-Butylphthalate	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
di-n-Octylphthalate - CCC	5.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Dibenz(a,h)anthracene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Dibenzofuran	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Diethylphthalate	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Dimethylphthalate	5.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Fluoranthene – CCC	1.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Fluorene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Hexachlorobenzene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Hexachlorobutadiene – CCC	1.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Hexachlorocyclopentadiene – SPCC	20	≤ 15	+ 30	<b>RF &gt; 0.050</b>	≤ 25%

Analyte	RL (µg)	Acceptance Criteria			
		ICAL (%RSD) <sup>①</sup>	ISCV (%R) <sup>②</sup>	CCV <sup>③</sup>	Precision\%RP F
Hexachloroethane	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Indeno(1,2,3-c,d)pyrene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Isophorone	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
n-Nitroso-di-n-propylamine – SPCC	1.0	≤ 15	+ 30	<b>RF &gt; 0.050</b>	≤ 25%
n-Nitrosodiphenylamine – CCC	10	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Naphthalene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Nitrobenzene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Pentachlorophenol – CCC	20	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Phenanthrene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%
Phenol – CCC	5.0	≤ 30	+ 30	%D ≤ 20%	≤ 25%
Pyrene	1.0	≤ 15	+ 30	%D ≤ 20%	≤ 25%

- ① Can use the mean RSD criterion of ≤ 15% as noted in par. 7.5.1.2.1 of SW-846, 8000B.  
 ② No more than 10% of the target compounds are allowed to exceed the limit.  
 ③ If %D for all CCC is less than or equal to 20%, then the CCV is assumed to be valid. If the CCCs are not included in the list of analytes for a project, then all analytes must meet the 20% D.

**Table 6.6.12 Surrogates**

Analyte	Accuracy <sup>④</sup> (% R)
2,4,6-Tribromophenol	10 – 123
2-Fluorobiphenyl	43 – 116
2-Fluorophenol	21 – 110
Nitrobenzene-d <sub>5</sub>	35 – 114
Phenol-d <sub>5</sub>	10 – 110
p-Terphenyl-d <sub>14</sub>	33 – 141

**Table 6.6.13 Internal Standards**

Analyte	Accuracy (% R)
Acenaphthene-d <sub>10</sub>	-50 to +100
Chrysene-d <sub>12</sub>	-50 to +100
1,4-Dichlorobenzene-d <sub>4</sub>	-50 to +100
Naphthalene-d <sub>8</sub>	-50 to +100
Perylene-d <sub>12</sub>	-50 to +100
Phenanthrene-d <sub>10</sub>	-50 to +100

- ④ The Surrogate limits are derived from USEPA CLP OLM 03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of liquid samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits.

**Table 6.6.14 Extracted Laboratory Control Spikes**

Analyte	Accuracy <sup>①</sup> (% R)
1,2,4-Trichlorobenzene	39 – 98
1,4-Dichlorobenzene	36 – 97
2,4-Dinitrotoluene	24 – 96
2-Chlorophenol	27 – 123
4-Chloro-3-methylphenol	23 – 97
4-Nitrophenol	10 – 80
Acenaphthene	46 – 118
N-Nitroso-di-n-propylamine	41 – 116
Pentachlorophenol	9 – 103
Phenol	12 – 110
Pyrene	26 – 127

**Table 6.6.15 Pre-Spike Surrogates**

Analyte	Accuracy <sup>②</sup> (%R)
Benzo(a)Pyrene-d <sub>12</sub>	50 – 150
Fluoranthene- d <sub>10</sub>	50 – 150

① The LCS limits are derived from USEPA CLP OLM03.0 and OLM04.2. Air Toxics Ltd. receives a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of in-house Control Limits. These limits only apply to samples that are extracted by Air Toxics Ltd. When sample extracts are sent to Air Toxics Ltd., limits of 50 - 150% are applied.

② The pre-spike Surrogates limits are arbitrary. Air Toxics Ltd. received a numerically insufficient number of samples for SW 8270C analysis to allow semi-annual updating of in-house control limits.

**Table 6.6.16 Summary of Calibration and QC Procedures SW-846 Modified Method 8270C**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Prior to calibration and at start of every 12 hrs.	SW-846 tuning criteria for Semi-volatiles analysis.	Correct problem then repeat tune.
Initial 5-Point Calibration	Prior to sample analysis.	ICAL criteria in Table 6-7.11.	Correct problem then repeat Initial Calibration.
Independent Source Calib. Ver. (ISCV)	All analytes – once per Initial Calibration.	At least 90% of the target compounds recoveries must be between 70 – 130%.	Determine the source of discrepancy between standards. Re-calibrate if needed.
Continuing Calibration Verification (CCV)	At the start of every clock, immediately after the DFTPP tune check.	<b>SPCCs:</b> RF ≥ 0.050 <b>CCC's:</b> %D ≤ 20%; Non-CCC's when CCC compounds are not requested %D ≤ 20%.	Investigate and correct the problem, up to and including re-calibration if necessary. High bias for one or more compounds associated with non-detects in the samples will not result in re-analysis.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Internal Standards (IS)	As each standard, blank, and sample is being aliquoted.	<p><b>For CCVs:</b> area counts within -50% to +100% from the most recent ICAL.</p> <p><b>For blanks, samples and non-CCV QC Checks:</b> Retention Times within <math>\pm 0.33</math> minutes (20 seconds) and area counts within -50% to +100% of the CCV.</p>	<p><b>For CCVs:</b> Investigate and correct the problem before proceeding with sample analysis.</p> <p><b>For blanks:</b> Inspect the system and re-analyze the blank.</p> <p><b>For samples and non-CCV QC:</b> Re-analyze the samples. If the criteria are not met a second time, dilute sample until IS meet criteria.</p>
Surrogates	With all samples and blanks prior to extraction.	See Table 6-7.12.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Extracted LCS	With each set of up to 20 extracted samples.	See LCS Criteria in Table 6-7.14.	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise narrate.
Laboratory Blank	With each set of up to 20 extracted samples.	Results less than laboratory RL.	Re-aliquot and re-analyze the extract to confirm the presence of the target compound. If it doesn't confirm, investigate and correct the problem before re-analyzing all the affected samples.
Solvent Blanks	When samples that are extracted together are analyzed on different analytical shifts.	All target compounds below the RL.	Investigate and correct the problem before re-analyzing all the affected samples.
Laboratory Duplicates	10% of the samples.	RPD $\leq 25\%$ for all detections $> 5 X$ 's RLs.	Analyze a third time. Report the closest two results and narrate and report the data if the criteria is still not met.

**6.7 TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS**

This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/TO-15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either concentrated using a cryogenic trap and/or concentrated using a hydrophobic multisorbent bed. The hydrophobic multisorbent bed functions as a drying system which removes water from the sample stream prior to analysis by full scan GC/MS. During analysis, the sample may be focused onto a cryogenic cooled column and/or a cryogenic cooled sleeve for analysis by full scan GC/MS.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.7.1 Summary of Method Modifications**

<b>Requirement</b>	<b>TO-14A</b>	<b>TO-15</b>	<b>Air Toxics Ltd. Modifications</b>
Sample Drying System	Nafion Drier.	Multisorbent.	Multisorbent.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blanks and standards ( <b>applies to Low Level analysis only</b> )	Zero Air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.
Initial Calibration	≤ 30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % for <b>QUAD and 5&amp;20</b> analysis and 4 compounds allowed out to ≤ 40 % for <b>Low Level</b> analysis.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Daily CCV	≤ 30% D.	≤ 30% D.	<p>For <b>QUAD and 5&amp;20</b> analysis: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.</p> <p>For <b>Low Level</b> analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.</p>
Sample collection media.	Summa canister.	Summa canister.	<p>Methods TO-14A/TO-15 are validated for samples collected in specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of these methods and not recommended for ambient or indoor air samples. Associated results are considered qualified.</p>

**Table 6.7.2 Method TO-14A/TO-15 Analyte List**

Analyte	RL (ppbv) LL/TO- 15/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,1,2-Trichloroethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethene	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,2,4-Trichlorobenzene	2.0/0.5/20	30%	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,2-Dichlorobenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,2-Dichloroethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,2-Dichloropropane	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,3-Dichlorobenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
1,4-Dichlorobenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Benzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Bromomethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
Carbon Tetrachloride	0.1/0.5/5.0	30%	70 - 130	≤ 25
Chlorobenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Chloroethane	0.1/0.5/5.0	30%	70 - 130	≤ 25
Chloroform	0.1/0.5/5.0	30%	70 - 130	≤ 25
Chloromethane	2.0/0.1/20	30%	70 - 130	≤ 25
α-Chlorotoluene (Benzyl Chloride)	0.1/0.5/5.0	30%	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.1/0.5/5.0	30%	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Dichloromethane	0.5/0.2/5.0	30%	70 - 130	≤ 25
Ethylbenzene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.1/0.5/5.0	30%	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.1/0.5/5.0	30%	70 - 130	≤ 25
Freon 114	0.1/0.5/5.0	30%	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.1/0.5/5.0	30%	70 - 130	≤ 25
Hexachlorobutadiene	2.0/0.5/20	30%	70 - 130	≤ 25
m,p-Xylene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Methyl Chloroform (1,1,1-Trichloroethane)	0.1/0.5/5.0	30%	70 - 130	≤ 25
o-Xylene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Styrene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Tetrachloroethene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Toluene	0.1/0.5/5.0	30%	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Trichloroethene	0.1/0.5/5.0	30%	70 - 130	≤ 25
Vinyl Chloride	0.1/0.5/5.0	30%	70 - 130	≤ 25

**Table 6.7.3 Method TO-14A/TO-15 Analyte List**

Analyte	RL (ppbv) TO-15/ LL/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits
1,3-Butadiene	0.5/0.1/5.0	30%	60 – 140	≤ 25
1,4-Dioxane	2.0/0.1/20	30%	60 – 140	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.5/0.1/5.0	30%	60 – 140	≤ 25
2-Hexanone	2.0/0.5/20	30%	60 – 140	≤ 25
4-Ethyltoluene	0.5/0.1/5.0	30%	60 – 140	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.5/0.1/5.0	30%	60 – 140	≤ 25
Acetone	2.0/0.5/20	30%	60 – 140	≤ 25
Bromodichloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Bromoform	0.5/0.1/5.0	30%	60 – 140	≤ 25
Carbon Disulfide	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cyclohexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Dibromochloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Ethanol	2.0/0.5/20	30%	60 – 140	≤ 25
Heptane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Hexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Isopropanol (2-Propanol)	2.0/0.5/20	30%	60 – 140	≤ 25
Methyl t-Butyl Ether (MTBE)	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylene	2.0/0.5/20	30%	60 – 140	≤ 25
Tetrahydrofuran	0.5/0.5/5.0	30%	60 – 140	≤ 25
trans-1,2-Dichloroethene	0.5/0.1/5.0	30%	60 – 140	≤ 25
2,2,4-Trimethylpentane	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cumene	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylbenzene	0.5/0.1/5.0	30%	60 – 140	≤ 25
3-Chloroprene	2.0/0.5/20	30%	60 – 140	≤ 25
Naphthalene	2.0/0.5/20	30%	60 – 140	≤ 25
TPH (Gasoline) or NMOC (Hexane/Heptane)	10/2.0/100	One Point Calibration	NA	≤ 25

**Table 6.7.4 Internal Standards**

**Table 6.7.5 Surrogates**

Analyte	Accuracy (% R)	Analyte	Accuracy (% R)
Bromochloromethane	60 - 140	1,2-Dichloroethane-d <sub>4</sub>	70 – 130
1,4-Difluorobenzene	60 - 140	Toluene-d <sub>8</sub>	70 – 130
Chlorobenzene-d <sub>5</sub>	60 - 140	4-Bromofluorobenzene	70 – 130

**Table 6.7.6 Summary of Calibration and QC Procedures for Methods TO-14A/TO-15**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis.	% RSD $\leq$ 30 with two compounds allowed out to $\leq$ 40% RSD for QUAD and 5&20 (4 allowed out for LL).	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be 70-130%; for 80% of "Non-standard" compounds, recoveries must be 60-140%. No recovery may be <50%.  * If specified by the client in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	For QUAD and 5&20: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.  For <b>Low Level</b> analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5 point calibration curve.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Blank	After the CCV/LCS.	Results less than the laboratory reporting limit.	Inspect the system and Re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within $\pm 0.33$ min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards.	<p><b>For blanks:</b> inspect the system and reanalyze the blank. Analysis is discontinued until the blank meets the IS criteria.</p> <p><b>For samples:</b> re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.</p>
Surrogates	As each standard, blank, and sample is being loaded.	<p>70 - 130%.</p> <p>* If specified by the client in-house generated control limits may be used.</p>	<p><b>For blanks:</b> inspect the system and reanalyze the blank. Analysis is discontinued until the blank meets the surrogate recovery criteria</p> <p><b>For samples:</b> re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.</p>
Laboratory Duplicates	10% of the samples.	RPD $\leq 25\%$ for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate results.

**6.8 TO-14A/TO-15 VOLATILE ORGANIC COMPOUNDS BY SIM**

This method involves Selective Ion Monitoring (SIM) GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/TO-15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and concentrated using a cryogenic trap. The focused air sample is then flash heated through a hydrophobic drying system that removes water from the sample stream. The sample is then focused onto a cryogenic cooled column prior to analysis by GC/MS in the (SIM) mode.

Some MSD's can be set to acquire both SIM and full scan data simultaneously. This generates two separate data files in the analytical software. One file contains full scan data following the operating procedures outlined in this SOP and the other contains SIM data following the procedures in SOP #38. This allows a lower reporting limit for the selected SIM compounds. The results for each sample in a report will be from two separate

data files originating from the same analytical run. The two data files have the same base file name and are differentiated with a "sim" extension on the SIM data file.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The target analyte list and Limit of Quantitation reflect relevant risk driving compounds and are available upon request. The method modifications, QC criteria, and QC summary may be found in the following tables.

**Table 6.8.1 Summary of Method Modifications**

<b>Requirement</b>	<b>TO-14A</b>	<b>TO-15</b>	<b>Air Toxics Ltd. Modifications</b>
Sampling/concentrator system	Nafion Drier.	Multi-sorbent concentrator.	Multi-sorbent concentrator
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blank and standards	Zero air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40% D.
ICAL %RSD acceptance criteria	< 30% RSD.	≤ 30%, with two compounds allowed to ≤ 40%.	Project specific; default criteria is ≤30% RSD with 10% of compounds allowed out to ≤ 40% RSD.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Daily CCV	≤30% D.	≤30% D.	Project specific; default criteria is 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated. If more than 10% of compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.

**Table 6.8.2 Internal Standards**

**Table 6.8.3 Surrogates**

<i>Analyte</i>	Accuracy (% R)	<i>Analyte</i>	Accuracy (% R)
Bromochloromethane	60 - 140	1,2-Dichloroethane-d <sub>4</sub>	70 - 130
1,4-Difluorobenzene	60 - 140	Toluene-d <sub>8</sub>	70 - 130
Chlorobenzene-d <sub>5</sub>	60 - 140	4-Bromofluorobenzene	70 - 130

**Table 6.8.4 Summary of Calibration and QC Procedures for Methods TO-14A/TO-15 by SIM**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW - 846 tune criteria.	Correct problem then repeat tune.
5-6-Point Calibration	Prior to sample analysis	≤ 30% for standard compounds with 10% of the compound list allowed out to ≤ 40% RSD.	Correct problem then repeat Initial Calibration Curve.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Laboratory Control Standard (LCS)	After each initial calibration curve, and daily prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be $\pm 30\%$ ; for 80% of "Non-standard" compounds, recoveries must be $\pm 40\%$ . No recovery may be $< 50\%$ .  * If specified by the client in-house generated control limits may be used.	Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than 10% of compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new calibration curve.
Laboratory Blank	After the LCS.	Results less than the laboratory reporting limit.	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within $\pm 0.33$ min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards.	<b>For blanks:</b> inspect the system and re-analyze the blank. <b>For samples:</b> re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%.  * If specified by the client in-house generated control limits may be used.	<b>For blanks:</b> inspect the system and re-analyze the blank. <b>For samples:</b> re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, report data from first analysis and narrate.

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Laboratory Duplicates	10% of the samples.	RPD $\leq$ 25% for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

**6.9 ASTM D- 1945 – FIXED GASES**

This method involves GC analysis of landfill gas, ambient air, or stack gas collected in Summa™ canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane and fixed gases and can be used to speciate individual light hydrocarbons up to C6. This method is also used to determine caloric content of the gas. Because the sample is withdrawn from the vessel by positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a FID and a Thermal Conductivity Detector (TCD).

Certain compounds are not included in ATL’s standard target analyte list. These compounds are communicated at the time of client

proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.9.1 Summary of Method Modifications**

<b>Requirement</b>	<b>ASTM D-1945</b>	<b>Air Toxics Ltd. Modifications</b>
Sample Injection Volume	0.50 mL to achieve Methane linearity.	1.0 mL.
Reference Standard	Concentration should not be < half of nor differ by more than 2X the concentration of the sample. Run 2 consecutive checks; must agree within 1%.	A minimum 3-point linear calibration. The acceptance criterion is RSD ≤15%. All target analytes must be within the linear range of calibration (with the exception of O <sub>2</sub> , N <sub>2</sub> , and C6+ Hydrocarbons).
Sample Analysis	Equilibrate samples to 20-50° F. above source temperature at field sampling.	Heating of samples is not performed.
Sample Calculation	Response factor is calculated using peak height for C5 and lighter compounds.	Peak areas are used for all target analytes to quantitate concentrations.
Normalization	Sum of original values Should not differ from 100.0% by more than 1.0%.	Sum of original values may range between 85-115%; normalization of data not performed, unless requested by client.

**Table 6.9.2 ASTM Modified Method D-1945 Standard Analyte List**

Analyte	RL (%)	Acceptance Criteria		
		Initial Calibration (%RSD)	CCV/LCS (%R)	Precision (%RPD)
Carbon Dioxide	0.01	≤ 15%	85 – 115	≤ 25%
Carbon Monoxide	0.01	≤ 15%	85 – 115	≤ 25%
Ethene	0.001	≤ 15%	85 – 115	≤ 25%
Ethane	0.001	≤ 15%	85 – 115	≤ 25%
Acetylene	0.001	≤ 15%	85 – 115	≤ 25%
Isobutane	0.001	≤ 15%	85 – 115	≤ 25%
Methane	0.0001	≤ 15%	85 – 115	≤ 25%
n-Butane	0.001	≤ 15%	85 – 115	≤ 25%
Neopentane	0.001	≤ 15%	85 – 115	≤ 25%
Isopentane	0.001	≤ 15%	85 – 115	≤ 25%
n-Pentane	0.001	≤ 15%	85 – 115	≤ 25%
Nitrogen*	0.10	≤ 15%	85 – 115	≤ 25%
NMOC (C6+)	0.01	≤ 15%	85 – 115	≤ 25%
Oxygen	0.10	≤ 15%	85 – 115	≤ 25%
Propane	0.001	≤ 15%	85 – 115	≤ 25%
Hydrogen	0.01***	≤ 15%	85 – 115	≤ 25%
Helium	0.01**	≤ 15%	85 – 115	≤ 25%

\* For samples that have been pressurized with N<sub>2</sub>, the amount of N<sub>2</sub> in the sample is determined by subtraction.

\*\* Included by special request only.

\*\*\* RL is 1.0% when sample is pressurized with Helium.

**Table 6.9.3 Summary of Calibration and QC Procedures ASTM Modified Method D-1945**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to Sample Analysis.	ICAL criteria in Table 6-10.2.	Correct problem, then repeat Initial Calibration.
Independent Source Check Verification (LCS)	Once per Initial Calibration and with each analytical batch.	LCS criteria in Table 6-10.2.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples or at the end of the analytical batch.	CCV criteria in Table 6-10.2.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met. If the closing CCV fails, the system is checked and the standard is re-analyzed. If the second analysis fails, identify and correct the problem, then re-analyze all samples since the last acceptable CCV.
Laboratory Blank	Daily.	Results less than the laboratory RL.	Inspect the system and troubleshoot until the system is free of contamination.
Sample Duplicates	10%.	RPD $\leq 25\%$ for detections $> 5$ times the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

**6.10 ASTM D-1946 - ATMOSPHERIC GASES**

This method involves GC analysis of landfill gas, ambient air, or stack gas collected in Summa™ canisters, Tedlar bags, or any vessel that has been demonstrated to be clean and leak free. Samples are analyzed for Methane, fixed gases, and Non-Methane Organic Carbon (NMOC) using ASTM D-1946 protocols. Because the sample is withdrawn from the vessel by positive pressure, rigid containers are first filled to positive pressure using UHP Helium or Nitrogen. Samples are then analyzed using a GC equipped with a FID and a TCD.

Certain compounds are not included in ATL's standard target analyte list. These compounds

are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.10.1 Summary of Method Modifications**

<b>Requirement</b>	<b>ASTM D-1946</b>	<b>Air Toxics Ltd. Modifications</b>
Calibration	A single point calibration is performed using a reference standard closely matching the composition of the unknown.	A minimum 3-point calibration curve is performed. Quantitation is based on a daily calibration standard, which may or may not resemble the composition of the associated samples.
Reference Standard	The composition of any reference standard must be known to within 0.01 mol % for any component.	The standards used by Air Toxics Ltd. are blended to a ≥ 95% accuracy.
Sample Injection Volume	Components whose concentrations are in excess of 5 % should not be analyzed by using sample volumes greater than 0.5 mL.	The sample container is connected directly to a fixed volume sample loop of 1.0 mL. Linear range is defined by the calibration curve. Bags may be loaded by vacuum or by positive pressure.
Normalization	Normalize the mole percent values by multiplying each value by 100 and dividing by the sum of the original values. The sum of the original values should not differ from 100% by more than 1.0%.	Results are not normalized (unless requested by client). The sum of the reported values can differ from 100% by as much as 15%, either due to analytical variability or an unusual sample matrix.
Precision	Precision requirements established at each concentration level.	Duplicates should agree within 25 % RPD for detections >5 X's the RL.

**Table 6.10.2 ASTM Modified Method D-1946 Standard Analyte List**

Compound	RL (%)	ICAL Criteria (%RSD)	LCS Criteria (%R)	CCV Criteria (%D)	Precision Limits (RPD)
Carbon Dioxide***	0.010	≤ 15%	85 – 115	±15	≤ 25%
Carbon Monoxide***	0.010	≤ 15%	85 – 115	±15	≤ 25%
Methane	0.00010	≤ 15%	85 – 115	±15	≤ 25%
Ethene*	0.0010	≤ 15%	85 – 115	±15	≤ 25%
Ethane*	0.0010	≤ 15%	85 – 115	±15	≤ 25%
Nitrogen	0.10	≤ 15%	85 – 115	±15	≤ 25%
NMOC (C2+)	0.010	≤ 15%	85 – 115	±15	≤ 25%
Oxygen	0.10	≤ 15%	85 – 115	±15	≤ 25%
Hydrogen*	0.010**	≤ 15%	85 – 115	±15	≤ 25%

\* Ethene, Ethane and Hydrogen are included by special request only.

\*\* RL is 1.0 % when sample is pressurized with He.

\*\*\* RL can be lowered to 0.001% using a Nickel catalyst and reporting from the FID by special request.

**Table 6.10.3 Summary of Calibration and QC Procedures ASTM Modified Method D-1946**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Initial Calibration Curve (ICAL)	Prior to Sample analysis.	RSD ≤ 15 %.	Correct problem then repeat Initial Calibration.
Second Source Verification (LCS)	All analytes - once per Initial Calibration, and with each analytical batch.	%R 85 – 115 %.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis and after every 20 samples.	%R 85 – 115 %.	Check the system and re-analyze the standard. Re-calibrate the instrument if the criteria cannot be met.
Laboratory Blank (He) (N <sub>2</sub> for He and H <sub>2</sub> analysis)	Immediately after each daily check standard and prior to sample analysis, or when contamination is present.	Results < RL.	Inspect the system and re-analyze the Blank.
End Check	At the end of analytical sequence. It can be primary (CCV) or second source (LCS).	%R 85 – 115 %.	Check system and re-analyze the standard. If the 2 <sup>nd</sup> analysis fails, correct the problem. Re-analyze all samples since the last acceptable CCV.
Sample Duplicates	10% of the samples.	RPD ≤ 25 % for detections > 5 X's the RL.	Re-analyze the sample a third time. Correct the problem. If no problem is found, narrate.

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Chromatographic Resolution of CH <sub>4</sub> from CO (FID)	As needed.	< 50 % valley.	Re-condition the molecular sieve column at similar levels.
Response of CO And CO <sub>2</sub> (FID)	As needed.	< 30 %.	Re-pack the tube with fresh catalyst and allow to stabilize.

**6.11 ASTM D-5504 - SULFUR COMPOUNDS**

This method involves GC analysis of whole air samples collected in Tedlar bags. Samples are analyzed for reduced sulfur compounds using ASTM D-5504 protocols using a Sulfur Chemiluminescence Detector (SCD).

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds

with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

ASTM D-5504 is not a prescriptive method therefore modification documentation is not necessary.

**Table 6.11.1 ASTM Modified Method D-5504 (Sulfur Compounds) Standard Analyte List**

Analyte	RL (ppbv)	Acceptance Criteria		
		ICAL <sup>①</sup> (% RSD)	LCS/ CCV <sup>②</sup> (% R)	Precision (% RPD)
2,5-Dimethylthiophene	4.0	≤ 30	70 -130	≤ 25
2-Ethylthiophene	4.0	≤ 30	70 -130	≤ 25
3-Methylthiophene**	4.0	≤ 30	70 -130	≤ 25
Carbon Disulfide	4.0	≤ 30	70 -130	≤ 25
Carbonyl Sulfide	4.0	≤ 30	70 -130	≤ 25
Diethyl Disulfide	4.0	≤ 30	70 -130	≤ 25
Diethyl Sulfide	4.0	≤ 30	70 -130	≤ 25
Dimethyl Disulfide	4.0	≤ 30	70 -130	≤ 25
Dimethyl Sulfide	4.0	≤ 30	70 -130	≤ 25
Ethyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Ethyl Methyl Sulfide**	4.0	≤ 30	70 -130	≤ 25
Hydrogen Sulfide	4.0	≤ 30	70 -130	≤ 25
Isobutyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Isopropyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Methyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
n-Butyl Mercaptan**	4.0	≤ 30	70 -130	≤ 25
n-Propyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
tert-Butyl Mercaptan	4.0	≤ 30	70 -130	≤ 25
Tetrahydrothiophene	4.0	≤ 30	70 -130	≤ 25
Thiophene	4.0	≤ 30	70 -130	≤ 25

<sup>①</sup> Average %RSD ≤ 30%, not to exceed 40% for any individual compounds. H<sub>2</sub>S %RSD must be ≤ 30%.

<sup>②</sup> Up to 10% allowed to exceed %R criterion (not to exceed ±50%); end check may have 20% exceed criterion. All compounds must be within %R limit for short list (five compounds or less)

\*\* Compounds co-elute

**Table 6.11.2 Summary of Calibration and QC Procedures for Modified ASTM Method D 5504**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Min of 3 or more points Calibration (ICAL)	Prior to sample analysis.	RSD $\leq$ 30% (average). H <sub>2</sub> S must be $\leq$ 30%. All others must be $\leq$ 40%.	Repeat calibration.
Second Source Verification (LCS)	With each Initial Calibration; with each analytical batch.	70 - 130 % of the expected values for at least 18 of the 20 target compounds. H <sub>2</sub> S must be within $\pm$ 30%. Recovery < 50% or > 150% will require corrective action. If less than five compounds, all compounds must meet criteria.	Check the system, re-prepare and/or re-analyze standard. Re-calibrate instrument if criteria cannot be met.
Continuing Calibration Verification (CCV)	Daily prior to sample analysis.	<b>Full list:</b> % R for at least 18 out of the 20 compounds within 70 – 130 %. H <sub>2</sub> S must meet limits. <b>Short list:</b> 5 compounds or less, % R for all compounds within 70 – 130 %. 5 – 19 compounds, %R for 1 compound or 10% of the compounds allowed out. H <sub>2</sub> S must meet limits.	Check the system and re-analyze the standard. If the 2 <sup>nd</sup> analysis fails, identify and correct the problem. Corrective action may include re-analysis of affected samples out of Hold Time per client request.
Laboratory Blank	In between analysis of standards and project samples.	Results less than the laboratory Limit of Quantitation.	Inspect the system and re-analyze the blank. If the third blank still has contamination, consult a Scientist or Department Manager.
End Check	At the end of the analytical sequence.	Recoveries within 70 - 130% with 20% (4 target analytes) allowed out.	Check system and re-analyze the standard. If the 2 <sup>nd</sup> analysis fails, identify and correct the problem. Corrective action may include re-analysis of affected samples out of Hold Time per client request.

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
Laboratory Duplicates	10% of the samples.	RPD $\leq$ 25 % for detections > 5X LOQ.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate the data.

**6.12 TO-17 VOLATILE ORGANIC COMPOUNDS**

This method is an alternative to the canister based sampling and analysis methods that are presented in EPA Compendium Methods TO-14 and TO-15. Samples are collected by drawing a volume of air through a sorbent packed tube. The sample cartridges are thermally desorbed by heating and purging with organic-free Helium. The resulting gaseous effluent is then bubbled through 5 ml of organic free reagent grade water and trapped on the sorbent trap of the purge and trap system or based on application may be trapped directly on a secondary trap. The sorbent or secondary trap is then thermally desorbed for GC/MS analysis.

The procedures in this method outline the use of EPA Method TO-17 protocols to determine the concentrations of volatile organic compounds in air samples collected on sorbent tubes.

Certain compounds are not included in ATL's standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.12.1 Summary of Method Modifications**

<b>Requirements</b>	<b>EPA Method TO-17</b>	<b>Air Toxics Ltd. Modifications</b>
Lab Blank	At least 2 tubes from the same cleaning batch as the samples are analyzed at the beginning and end of the analytical sequence.  Do not dry purge Lab Blanks.	Tubes used for daily lab blank may or may not be from the same batch or sampling media. Only 1 lab blank is analyzed prior to sample analysis. Lab blanks are dry purged to eliminate the possibility of sample anomaly attributed to Dry purge process.
*Tune Check	BFB.	Modification applies only to semivolatile lists such as PAHs in which a DFTPP tune check is more appropriate to demonstrate accurate spectral performance.
*Sample desorption	Method involves primary and secondary desorption.	Modification applies only when using a Tekmar P&T system. After primary desorption, the stream of effluent gas is passed through 5ml of clean purged D.I. water before the secondary desorption. D.I. water acts as a filter for excessive acidic moisture in the samples.

\*Modifications are dependent on application.

**Table 6.12.2 Summary of Sorbent Applications**

Sorbent	Typical Analyte Range	Water management	Primary Applications
Carbotrap 300	C3 – C12	High levels of moisture may interfere with analysis.	Indoor air and outdoor air.
Tenax TA	C7 – C26	Hydrophobic.	All vapors including soil gas.
Tenax GR	C7 – C30	Hydrophobic.	All vapors including soil gas.
Tenax TA/ Carbograph 1/Carbograph 5	C4-C26	Hydrophobic.	All vapors including soil gas.

**Table 6.12.3 TO-17 Carbotrap 300 Analyte List**

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV (%D)
1,1,1-Trichloroethane	10	30	70 – 130	30
1,1,1,2-Tetrachloroethane	10	30	70 – 130	30
1,1,2,2-Tetrachloroethane	10	30	70 – 130	30
1,1,2-Trichloroethane	10	30	70 – 130	30
1,1-Dichloroethane	10	30	70 – 130	30
1,1-Dichloroethene	10	30	70 – 130	30
1,1-Dichloropropene	10	30	70 – 130	30
1,2,3-Trichlorobenzene	50	30	70 – 130	30
1,2,3-Trichloropropane	10	30	70 – 130	30
1,2,4-Trichlorobenzene	50	30	70 – 130	30
1,2,4-Trimethylbenzene	10	30	70 – 130	30
1,2-Dibromo-3-chloropropane	50	30	70 – 130	30
1,2-Dichlorobenzene	10	30	70 – 130	30
1,2-Dichloroethane	10	30	70 – 130	30
1,2-Dichloropropane	10	30	70 – 130	30
1,3,5-Trimethylbenzene	10	30	70 – 130	30
1,3-Butadiene	50	30	50 – 150	30
1,3-Dichlorobenzene	10	30	70 – 130	30
1,3-Dichloropropane	10	30	70 – 130	30
1,4-Dichlorobenzene	10	30	70 – 130	30
2,2-Dichloropropane	50	30	70 – 130	30
2-Chloropropane	50	30	70 – 130	30
2-Chlorotoluene	10	30	70 – 130	30
Allyl chloride	50	30	70 – 130	30
4-Chlorotoluene	10	30	70 – 130	30
Acrylonitrile	50	30	70 – 130	30
Benzene	10	30	70 – 130	30
Bromobenzene	10	30	70 – 130	30
Bromochloromethane	10	30	70 – 130	30

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV (%D)
Bromodichloromethane	10	30	70 – 130	30
Bromoform	10	30	70 – 130	30
Bromomethane	10	30	50 – 150	30
Butylbenzene	10	30	70 – 130	30
Carbon Disulfide	10	30	70 – 130	30
Carbon Tetrachloride	10	30	70 – 130	30
Chlorobenzene	10	30	70 – 130	30
Chloroethane	10	30	50 – 150	30
Chloroform	10	30	70 – 130	30
Chloromethane	10	30	50 – 150	30
cis-1,2-Dichloroethene	10	30	70 – 130	30
cis-1,3-Dichloropropene	10	30	70 – 130	30
cis-1,4-Dichloro-2-butene	50	30	70 – 130	30
Cumene	10	30	70 – 130	30
Dibromochloromethane	10	30	70 – 130	30
Dibromomethane	10	30	70 – 130	30
Dichlorodifluoromethane	10	30	50 – 150	30
Ethylbenzene	10	30	70 – 130	30
Ethylene Dibromide	10	30	70 – 130	30
Freon 11	10	30	70 – 130	30
Freon 113	10	30	70 – 130	30
Hexachlorobutadiene	50	30	70 – 130	30
Hexane	10	30	70 – 130	30
Iodomethane	50	30	70 – 130	30
Methylene Chloride	10	30	70 – 130	30
Methyl t-butyl ether (MTBE)	10	30	70 – 130	30
Naphthalene	50	30	70 – 130	30
m,p-Xylene	10	30	70 – 130	30
o-Xylene	10	30	70 – 130	30
p-Cymene	10	30	70 – 130	30
Propylbenzene	10	30	70 – 130	30
sec-Butylbenzene	10	30	70 – 130	30
Styrene	10	30	70 – 130	30
tert-Butylbenzene	10	30	70 – 130	30
Tetrachloroethene	10	30	70 – 130	30
Toluene	10	30	70 – 130	30
trans-1,2-Dichloroethene	10	30	70 – 130	30
trans-1,3-Dichloropropene	10	30	70 – 130	30
trans-1,4-Dichloro-2-butene	50	30	70 – 130	30
Trichloroethene	10	30	70 – 130	30
Vinyl Bromide *	50	30	50 – 150	30

Analytes	RL (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV (%D)
Vinyl Chloride	10	30	50 – 150	30

\* Independent Source Verification Check not available for this compound.

Table 6.12.4 Internal Standard Recovery Limits (Carbotrap 300)

Analyte	CCV IS (%R)	Sample IS (%R)
1,4-Dichlorobenzene-d <sub>4</sub>	50 – 200	60 – 140
Chlorobenzene-d <sub>5</sub>	50 – 200	60 – 140
Fluorobenzene	50 – 200	60 – 140

Table 6.12.5 Field Surrogate Recovery Limits (Carbotrap 300)

Analyte	Accuracy (%R)
Benzene-d <sub>6</sub>	50 – 150
4-Bromofluorobenzene	70 – 130
Naphthalene-d <sub>8</sub>	70 – 130
Toluene-d <sub>8</sub>	70 – 130

Table 6.12.6 TO-17 (Tenax GR/TA)

Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
1,1,1-Trichloroethane	5.0	30	70 – 130	30
1,1,1,2-Tetrachloroethane	5.0	30	70 – 130	30
1,1,2,2-Tetrachloroethane	5.0	30	70 – 130	30
1,1,2-Trichloroethane	5.0	30	70 – 130	30
1,1-Dichloropropene	5.0	30	70 – 130	30
1,2,3-Trichlorobenzene	5.0	30	70 – 130	30
1,2,3-Trichloropropane	5.0	30	70 – 130	30
1,2,4-Trichlorobenzene	5.0	30	70 – 130	30
1,2,4-Trimethylbenzene	5.0	30	70 – 130	30
1,2-Dibromo-3-chloropropane	5.0	30	70 – 130	30
1,2-Dichlorobenzene	5.0	30	70 – 130	30
1,2-Dichloroethane	5.0	30	70 – 130	30
1,2-Dichloropropane	5.0	30	70 – 130	30
1,3,5-Trimethylbenzene	5.0	30	70 – 130	30
1,3-Dichlorobenzene	5.0	30	70 – 130	30
1,3-Dichloropropane	5.0	30	70 – 130	30
1,4-Dichlorobenzene	5.0	30	70 – 130	30
2-Chlorotoluene	5.0	30	70 – 130	30
4-Chlorotoluene	5.0	30	70 – 130	30
Benzene	5.0	30	70 – 130	30
Bromobenzene	5.0	30	70 – 130	30
Bromodichloromethane	5.0	30	70 – 130	30
Bromoform	5.0	30	70 – 130	30
Butylbenzene	5.0	30	70 – 130	30
Carbon Tetrachloride	5.0	30	70 – 130	30
Chlorobenzene	5.0	30	70 – 130	30

Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Styrene	5.0	30	70 – 130	30
tert-Butylbenzene	5.0	30	70 – 130	30
Tetrachloroethene	5.0	30	70 – 130	30
Toluene	5.0	30	70 – 130	30
trans-1,3-Dichloropropene	5.0	30	70 – 130	30
trans-1,4-Dichloro-2-butene	5.0	30	70 – 130	30
Trichloroethene	5.0	30	70 – 130	30
Chloroform	5.0	30	70 – 130	30
cis-1,3-Dichloropropene	5.0	30	70 – 130	30
cis-1,4-Dichloro-2-butene	5.0	30	70 – 130	30
Cumene	5.0	30	70 – 130	30
Dibromochloromethane	5.0	30	70 – 130	30
Dibromomethane	5.0	30	70 – 130	30
Ethylbenzene	5.0	30	70 – 130	30
Ethylene Dibromide	5.0	30	70 – 130	30
Hexachlorobutadiene	5.0	30	70 – 130	30
Naphthalene	5.0	30	70 – 130	30
m,p-Xylene	10	30	70 – 130	30
o-Xylene	5.0	30	70 – 130	30
p-Cymene	5.0	30	70 – 130	30
Propylbenzene	5.0	30	70 – 130	30
sec-Butylbenzene	5.0	30	70 – 130	30

**Table 6.12.7 Internal Standard Recovery Limits (Tenax GR/TA)**

Analyte	CCV IS (%R)	Sample IS (%R)
1,4-Dichlorobenzene-d <sub>4</sub>	50 – 200	60 – 140
Chlorobenzene-d <sub>5</sub>	50 – 200	60 – 140
Fluorobenzene	50 – 200	60 – 140

**Table 6.12.8 Field Surrogate Recovery Limits (Tenax GR/TA)**

Analyte	Accuracy (%R)
Benzene-d <sub>6</sub>	50 – 150
4-Bromofluorobenzene	70 – 130
Toluene-d <sub>8</sub>	70 – 130
Naphthalene-d <sub>8</sub> (optional)	70 – 130

**Table 6.12.9 TO-17 TPH External Calibration (Tenax GR/TA)**

Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Mineral Spirits (C9 – C12 range)	500	30	70 – 130	30
<b>Surrogates (optional)</b>		<b>% Recovery</b>		
Chlorobenzene-d5		70 – 140		
Naphthalene – d8		70 - 140		
Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Diesel	1000	30	70 – 130	30
Gasoline	1000	30	70 – 130	30
Kerosene	1000	30	70 – 130	30
<b>Surrogates (optional)</b>		<b>% Recovery</b>		
Toluene-d8		70 – 140		
4-Bromofluorobenzene		70 - 140		
Naphthalene – d8		70 - 140		

**Table 6.12.10 TO-17 (Tenax TA - Passive)**

Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Benzene	5.0	30	70 – 130	30
Toluene	5.0	30	70 – 130	30
Ethyl benzene	1.0	30	70 – 130	30
m,p-xylene	2.0	30	70 – 130	30
o-Xylene	1.0	30	70 – 130	30
Trichloroethene	1.0	30	70 – 130	30
Tetrachloroethene	1.0	30	70 – 130	30
Cis-1,2-Dichloroethene	1.0	30	70 – 130	30
Trans-1,2-Dichloroethene	1.0	30	70 – 130	30
1,1-Dichloroethene	1.0	30	70-130	30
<b>Internal Standards</b>				
Analyte	CCV IS % Recovery		Sample IS % Recovery	
1,4-Dichlorobenzene-d4	50 – 200		60 – 140	
Chlorobenzene-d5	50 – 200		60 – 140	
Fluorobenzene	50 – 200		60 – 140	
<b>Surrogates</b>				
Analyte	% Recovery			
1,2-Dichloroethane-d4	70 – 130			
4-Bromofluorobenzene	70 – 130			
Dibromofluoromethane	70 – 130			

**Table 6.12.11 TO-17 (Tenax GR-SVOC)**

Analytes	Reporting Limit (ng)	Acceptance Criteria		
		ICAL (%RSD)	LCS (% R)	CCV
Naphthalene	5.0	30	70 – 130	30
2-Methylnaphthalene	5.0	30	70 – 130	30
Acenaphthylene	5.0	30	70 – 130	30
Acenaphthene	5.0	30	70 – 130	30
Fluorene	5.0	30	70 – 130	30
Phenanthrene	5.0	30	70 – 130	30
Anthracene	5.0	30	70 – 130	30
Fluoranthene	5.0	30	70 – 130	30
Pyrene	10	30	70 – 130	30
Internal Standards				
Analyte	CCV IS % Recovery		Sample IS % Recovery	
Naphthalene-d8	50 – 200		60 – 140	
Acenaphthene-d10	50 – 200		60 – 140	
Phenanthrene-d10	50 – 200		60 – 140	
Surrogates				
Analyte	% Recovery			
Fluorene-d10	70 – 130			
Pyrene-d10	70 – 130			

**Table 6.12.12 Summary of Calibration and QC Procedures for Method TO-17 (Volatile Organic Compounds)**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW - 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample Analysis.	%RSD ≤ 30%, 2 allowed out up to 40%	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial Calibration Curve and daily prior to analysis.	Recovery 70- 130% or 50- 150% as noted in Table 6-14.3.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	70 - 130 %	If project specified risk drivers exceed this criteria, more than 5% of the compounds exceed this criteria, or any VOC exceeds 50-150% recovery, maintenance is performed and the CCV test repeated. If the system still fails the CCV, perform a new 5-point Calibration Curve.
Laboratory Blank	After the CCV.	Results less than the RL.	Inspect the system and re-analyze the Blank.
Internal Standard (IS)	As each standard, Blank, and sample is being loaded.	<p><b>CCVs:</b> area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL.</p> <p><b>Blanks and samples:</b> Retention time (RT) must be within <math>\pm 0.33</math> minutes of the RT in the CCV. The IS area must be within <math>\pm 40\%</math> of the CCV's IS area for the Blanks and samples.</p>	<p><b>CCV:</b> inspect and correct system prior to sample analysis.</p> <p><b>Blanks:</b> inspect the system and re-analyze the Blank.</p> <p><b>Samples:</b> samples cannot be re-analyzed due to the nature of the sorbent cartridges. However investigate the problem by reviewing the data. If necessary, run a Lab Blank to check the instrument performance. Report the data and narrate.</p>
Surrogates	As each standard, Blank, and sample is being loaded.	70 - 130%.	<p><b>For blanks:</b> inspect the system and re-analyze the Blank.</p> <p><b>For samples:</b> samples cannot be re-analyzed due to the nature of sorbent cartridges. However investigate the problem by reviewing the data. If necessary, run a Lab Blank to check the instrument performance. Report the data and narrate the problem.</p>

**6.13 PASSIVE SAMPLING -  
VOLATILE ORGANIC  
COMPOUNDS**

This method involves GC/MS analysis of VOCs collected using charcoal-based passive samplers. These samplers are used to measure vapor-phase VOCs in a variety of gaseous matrices including indoor air, outdoor air, extracted soil gas, and emissions from materials. VOCs in the sampling environment pass through the diffusive barrier or permeable membrane of the sampler at a known, controlled rate (defined as the sampling rate) and adsorb to the charcoal-based sorbent pad of the sampler. The sorbent is extracted using a volume of carbon disulfide, and the extract is directly injected into a gas chromatograph equipped with a mass spectrometer. The retention time and spectral pattern of a compound is compared with that of known standard. Concentrations of the analytes are calculated from the average relative response

factors of calibration curves obtained from analysis of standard solutions. The results are reported in units of  $\mu\text{g/sample}$  or  $\mu\text{g/m}^3$  if the sampling rate and duration is known.

There are no regulatory methods for the preparation and analysis of the Radiello, PDMS, and 3M OVM 3500 samplers. The manufacturer of Radiello (FSM) and 3M have published recommended procedures which serve as the basis for this standard operating procedure. Additionally, QC elements outlined in EPA SW-846 8260 and 8270 are incorporated as applicable. One variance of note that ATL has taken to the Radiello method and the OVM 3500 method is the use of GC/MS instead of GC/FID.

**Table 6.13.1 Target Analytes**

\* Acceptance limits based on Desorption efficiency studies. \*\* 60 – 130% for PDMS

Analytes	Reporting Limit (µg/mL)	Acceptance Criteria			
		ICAL (%RSD)	ICV (% R)	LCS (% R)	CCV
Chloromethane	0.1	30	70 – 130	70 – 130	%D ≤30%
Vinyl Chloride	0.1	30	70 – 130	70 – 130	%D ≤30%
Ethanol	0.5	30	70 – 130	50 – 130*	%D ≤30%
1,1-Dichloroethene	0.1	20	80 – 120	80 – 120	%D ≤20%
Acetone	0.1	30	70 – 130	70 – 130	%D ≤30%
2-Propanol	0.1	30	70 – 130	70 – 130	%D ≤30%
MTBE	0.05	30	70 – 130	70 – 130	%D ≤30%
trans-1,2-Dichloroethene	0.1	20	80 – 120	80 – 120	%D ≤20%
Hexane	0.05	30	70 – 130	70 – 130	%D ≤30%
1,1-Dichloroethane	0.05	20	80 – 120	80 – 120	%D ≤20%
Ethyl Acetate	0.1	30	70 – 130	70 – 130	%D ≤30%
2-Butanone	0.05	30	70 – 130	70 – 130	%D ≤30%
cis-1,2-Dichloroethene	0.05	20	80 – 120	80 – 120	%D ≤20%
Chloroform	0.05	20	80 – 120	80 – 120	%D ≤20%
Cyclohexane	0.05	20	80 – 120	80 – 120	%D ≤20%
1,1,1-trichloroethane	0.05	20	80 – 120	80 – 120	%D ≤20%
Carbon Tetrachloride	0.05	20	80 – 120	80 – 120	%D ≤20%
Benzene	0.1	30	70 – 130	70 – 130	%D ≤30%
1,2-Dichloroethane	0.05	20	80 – 120	80 – 120	%D ≤20%
Heptane	0.05	20	80 – 120	80 – 120	%D ≤20%
Trichloroethene	0.05	20	80 – 120	80 – 120	%D ≤20%
2-Chloroethyl Vinyl Ether	1.0	30	70 – 130	20-130*	%D ≤30%
4-Methyl-2-pentanone	0.1	30	70 – 130	70 – 130	%D ≤30%
Toluene	0.05	20	80 – 120	80 – 120	%D ≤20%
1,1,2-Trichloroethane	0.05	20	80 – 120	80 – 120	%D ≤20%
Tetrachloroethene	0.05	20	80 – 120	80 – 120	%D ≤20%
Chlorobenzene	0.05	20	80 – 120	80 – 120	%D ≤20%
Ethylbenzene	0.05	20	80 – 120	80 – 120	%D ≤20%
m,p-Xylene	0.05	20	80 – 120	80 – 120	%D ≤20%
o-Xylene	0.05	20	80 – 120	80 – 120	%D ≤20%
Styrene	0.05	30	70 – 130	20-130*	%D ≤30%
1,1,2,2-Tetrachloroethane	0.05	30	70 – 130	70 – 130	%D ≤30%
Propylbenzene	0.05	20	80 – 120	80 – 120	%D ≤20%
1,3,5-Trimethylbenzene	0.05	20	80 – 120	80 – 120	%D ≤20%
1,2,4-Trimethylbenzene	0.05	20	80 – 120	80 – 120	%D ≤20%
1,3-Dichlorobenzene	0.05	30	70 – 130	70 – 130**	%D ≤30%
1,4-Dichlorobenzene	0.05	30	70 – 130	70 – 130**	%D ≤30%
1,2-Dichlorobenzene	0.05	30	70 – 130	70 – 130**	%D ≤30%
1,4-Dithian	0.05	30	70 – 130	70 – 130	%D ≤30%
Naphthalene	0.05	30	70 – 130	20-130*	%D ≤30%

**Table 6.13.2 Internal Standard**

<b>Analyte</b>	<b>CCV IS (%R)</b>	<b>Sample IS (%R)</b>
2-Fluorotoluene	50 – 200	50 – 200

**Table 6.13.3 Surrogate**

<b>Analyte</b>	<b>%R</b>
Toluene-d <sub>8</sub>	70-130

**Table 6.13.4 Sampling Rates for “Standard” target compounds (RAD 130)**

<b>Analytes</b>	<b>Reporting Limit (µg/mL )</b>	<b>Sampling Rates for Radiello 130 Sampler</b>
Chloromethane	0.1	Estimated
Vinyl Chloride	0.1	Estimated
<b>Ethanol</b>	<b>0.5</b>	<b>102</b>
1,1-Dichloroethene	0.1	Estimated
<b>Acetone</b>	<b>0.1</b>	<b>77</b>
<b>2-Propanol</b>	<b>0.1</b>	<b>52</b>
<b>MTBE</b>	<b>0.05</b>	<b>65</b>
trans-1,2-Dichloroethene	0.1	Estimated
<b>Hexane</b>	<b>0.05</b>	<b>66</b>
1,1-Dichloroethane	0.05	Estimated
<b>Ethyl Acetate</b>	<b>0.1</b>	<b>78</b>
<b>2-Butanone</b>	<b>0.05</b>	<b>79</b>
cis-1,2-Dichloroethene	0.05	Estimated
<b>Chloroform</b>	<b>0.05</b>	<b>75</b>
<b>Cyclohexane</b>	<b>0.05</b>	<b>54</b>
1,1,1-trichloroethane	0.05	Estimated
<b>Carbon Tetrachloride</b>	<b>0.05</b>	<b>67</b>
<b>Benzene</b>	<b>0.1</b>	<b>80</b>
<b>1,2-Dichloroethane</b>	<b>0.05</b>	<b>77</b>
<b>Heptane</b>	<b>0.05</b>	<b>58</b>
<b>Trichloroethene</b>	<b>0.05</b>	<b>69</b>
2-Chloroethyl Vinyl Ether	1.0	Estimated
<b>4-Methyl-2-pentanone</b>	<b>0.1</b>	<b>67</b>
<b>Toluene</b>	<b>0.05</b>	<b>74</b>
1,1,2-Trichloroethane	0.05	Estimated
<b>Tetrachloroethene</b>	<b>0.05</b>	<b>59</b>
<b>Chlorobenzene</b>	<b>0.05</b>	<b>68</b>
<b>Ethylbenzene</b>	<b>0.05</b>	<b>68</b>
<b>m,p-Xylene</b>	<b>0.05</b>	<b>70</b>
<b>o-Xylene</b>	<b>0.05</b>	<b>65</b>
<b>Styrene</b>	<b>0.05</b>	<b>61</b>
1,1,2,2-Tetrachloroethane	0.05	Estimated
<b>Propylbenzene</b>	<b>0.05</b>	<b>57</b>
1,3,5-Trimethylbenzene	0.05	Estimated
<b>1,2,4-Trimethylbenzene</b>	<b>0.05</b>	<b>50</b>
1,3-Dichlorobenzene	0.05	Estimated
<b>1,4-Dichlorobenzene</b>	<b>0.05</b>	<b>51</b>
1,2-Dichlorobenzene	0.05	Estimated
1,4-Dithian	0.05	Estimated
<b>Naphthalene</b>	<b>0.05</b>	<b>25</b>

**Table 6.13.5 Sampling Rates for “Standard” target compounds (OVM)**  
*Varied sampling rates for each compound (1 to 8hr) see the OVM Technical bulletin*

Analytes	Reporting Limit (µg/mL )	Sampling Rates for OVM Sampler
Chloromethane	0.1	Estimated
Vinyl Chloride	0.1	41
<b>Ethanol</b>	<b>0.5</b>	<b>44</b>
1,1-Dichloroethene	0.1	Estimated
<b>Acetone</b>	<b>0.1</b>	<b>40</b>
<b>2-Propanol</b>	<b>0.1</b>	<b>39</b>
<b>MTBE</b>	<b>0.05</b>	<b>38</b>
trans-1,2-Dichloroethene	0.1	Estimated
<b>Hexane</b>	<b>0.05</b>	<b>32</b>
1,1-Dichloroethane	0.05	33
<b>Ethyl Acetate</b>	<b>0.1</b>	<b>34</b>
<b>2-Butanone</b>	<b>0.05</b>	<b>36</b>
cis-1,2-Dichloroethene	0.05	Estimated
<b>Chloroform</b>	<b>0.05</b>	<b>34</b>
<b>Cyclohexane</b>	<b>0.05</b>	<b>32</b>
1,1,1-trichloroethane	0.05	31
<b>Carbon Tetrachloride</b>	<b>0.05</b>	<b>30</b>
<b>Benzene</b>	<b>0.1</b>	<b>80</b>
<b>1,2-Dichloroethane</b>	<b>0.05</b>	<b>33</b>
<b>Heptane</b>	<b>0.05</b>	<b>29</b>
<b>Trichloroethene</b>	<b>0.05</b>	<b>31</b>
2-Chloroethyl Vinyl Ether	1.0	Estimated
<b>4-Methyl-2-pentanone</b>	<b>0.1</b>	<b>30</b>
<b>Toluene</b>	<b>0.05</b>	<b>31</b>
1,1,2-Trichloroethane	0.05	30
<b>Tetrachloroethene</b>	<b>0.05</b>	<b>28</b>
<b>Chlorobenzene</b>	<b>0.05</b>	<b>29</b>
<b>Ethylbenzene</b>	<b>0.05</b>	<b>27</b>
<b>m,p-Xylene</b>	<b>0.05</b>	<b>27</b>
<b>o-Xylene</b>	<b>0.05</b>	<b>27</b>
<b>Styrene</b>	<b>0.05</b>	<b>29</b>
1,1,2,2-Tetrachloroethane	0.05	28
<b>Propylbenzene</b>	<b>0.05</b>	Estimated
1,3,5-Trimethylbenzene	0.05	Estimated
<b>1,2,4-Trimethylbenzene</b>	<b>0.05</b>	Estimated
1,3-Dichlorobenzene	0.05	Estimated
<b>1,4-Dichlorobenzene</b>	<b>0.05</b>	Estimated
1,2-Dichlorobenzene	0.05	Estimated
1,4-Dithian	0.05	Estimated
<b>Naphthalene</b>	<b>0.05</b>	<b>25</b>

**Table 6.13.6 Summary of Calibration and QC Procedure**

*Note: These criteria are used specifically for the standard list of analytes listed in Table 6.13.1.*

<b>QC Check</b>	<b>Minimum Frequency</b>	<b>Acceptance Criteria</b>	<b>Corrective Action</b>
<b>Tuning Criteria</b>	Prior to calibration and at the start of every 12-hour clock.	Method 8260B tuning criteria.	Correct problem then repeat tune.
<b>Initial 5-Point Calibration</b>	Prior to sample analysis.	Compound criteria in Table A-1.	Correct problem then repeat initial calibration.
<b>Initial Calibration Verification (ICV)</b>	Once per initial calibration.	See Table A-1	Verify concentrations and standard preparation.
<b>Continuing Calibration Verification (CCV)</b>	At the start of every shift immediately after the BFB tune check.	See "CCV criteria" column in Table A-1	Investigate and correct the problem, up to and including recalibration if necessary.
<b>Internal Standards (IS)</b>	IS is added at the time of extraction to all samples and QC samples.	<b>For CCVs:</b> area counts 50% - 200%, RT w/in 30 sec of mid-point in ICAL. <b>For blanks, samples and non-CCV QC Checks:</b> area counts 50 - 200%, RT w/in 20 sec. of RT in CCV.	<b>CCV:</b> inspect and correct system prior to sample analysis. <b>For blanks:</b> inspect the system and re-analyze the blank. <b>For samples:</b> re-analyze; if out again, flag data.
<b>Surrogate</b>	Surrogate is added at the time of extraction to all samples and QC samples.	See Table A-3.	Same as for Internal Standards.
<b>Solvent Blanks</b>	Immediately after the calibration standard or after samples with high concentrations	Results less than laboratory reporting limit (see Table A-1).	Re-aliquot and re-analyze solvent blank. If detections remain, flag concentrations in associated samples.
<b>Extracted Laboratory Blank</b>	Each set of up to 20 samples	Results less than the reporting limit.	Flag sample concentrations in associated extraction batch.
<b>Extracted LCS</b>	Each set of up to 20 samples.	See Table A-1	Re-aliquot and re-analyze the extract. If within limits, report the re-analysis. Otherwise, narrate.

**6.14 PM10/TSP**

This method involves equilibrating quartz filters in a conditioning environment of a specified temperature and humidity range and weighing the filters before and after field sampling. Samples are analyzed for PM10 using 40 CFR Part 50 Appendix J or for Total Suspended Particulate (TSP) using 40 CFR Part 50 Appendix B. An analytical balance

with 0.1 mg resolution is used to measure the filter weights. The corresponding change in mass represents the TSP or PM<sub>10</sub> result, expressed in µg or µg/m<sup>3</sup>. The reporting limit is typically 1000 µg. Sampling volumes are required to calculate in units of µg/m<sup>3</sup>.

**Table 6.14.1 Conditioning Environment Criteria**

Method	Conditioning Environment Temperature (°F)	Conditioning Environment Relative Humidity (%)
PM10	59°F – 86°F ± 5°F	20% – 45% ± 5%
TSP	59°F – 86°F ± 5°F	≤ 50% ± 5%

**Table 6.14.2 Summary of Calibration and QC Procedures for Methods PM10 and TSP**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Calibration	Calibration checks of 3.00 g and 5.00 g are weighed to bracket the expected filter weight of ~4.5 g prior to sample analysis and at the end of the analytical batch.	Accuracy limits of 3.00 g weight: 2.997 g – 3.003 g Accuracy limits of 5.00 g weight: 4.995 g - 5.005 g	Correct problem then repeat calibration.
Laboratory Duplicates	Unexposed Filters: one per analytical batch. Exposed Filters: one duplicate per workorder.	Unexposed Filters: Weights of the clean filters should be within ±0.0028 g of the original value. Exposed Filters: ≤ 25% RPD and weights must be within ±0.005 g.	Re-condition the filter and re-weigh.
Laboratory Blanks	Immediately after the calibration checks.	Post weight of Lab Blank is less than pre weight and the difference is < 0.0028 g.	Confirm the weight difference and narrate.

**6.15 AIR PHASE PETROLEUM  
HYDROCARBONS (MA APH)**

The MADEP APH method describes techniques for the analysis of air-phase petroleum hydrocarbons (APH) collected as whole air samples in stainless steel canisters. Up to 0.2 Liters of air is withdrawn from the canister through a mass flow controller and is cryofocused on a sorbent via liquid Nitrogen. The focused air sample is then flash heated

through a hydrophobic drying system which removes water from the sample stream prior to analysis by full scan GC/MS. Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 6.15.1 Summary of the Method Modifications**

<b>Requirement</b>	<b>APH</b>	<b>ATL Modifications</b>
Sample concentration/ Moisture Control	Cryogenically concentrated/Nafion dryer	Multibed sorbent
GC Column	RTX-1 60m*0.25mm*1um	RTX-1 60m*0.25mm*1um Or equivalent capillary column
Load volume	Maximum of 250 mL	Maximum of 200 mL
Sample pressurization	Zero Air or UHP Nitrogen	UHP Nitrogen
MDL study for Hydrocarbon Ranges	Gasoline standard is analyzed to determine MDL.	The calibration standard containing the aliphatic and aromatic compounds is analyzed.
Internal standard	Internal Standard should be a nominal 10ppbv or 10ug/m3	Internal standard is at 25ppbv
Laboratory Control Spike	Analyzed after the ICAL, specified subset is controlled. Recovery must be 70-130%.	Analyzed after the ICAL and daily; allow 10% of subset outside 70-130%.
Surrogates	Not required	Added surrogate

**Table 6.15.2 APH Target Compound List**

Analyte	Reporting Limit (ug/m3)	Acceptance Criteria	
		Accuracy Limits (%R)	Precision Limits (RPD)
1,3-Butadiene*	2.0	70 - 130	± 25
Methyl-tert-butyl ether (MTBE)*	2.0	70 - 130	± 25
Benzene*	2.0	70 - 130	± 25
Toluene*	2.0	70 - 130	± 25
Ethyl benzene*	2.0	70 - 130	± 25
m/p-Xylene*	2.0	70 - 130	± 25
o-Xylene*	2.0	70 - 130	± 25
Naphthalene	2.0	70 - 130	± 25

\*Compounds comprise the LCS/2<sup>nd</sup> Source Standard.

**Table 6.15.3 Aliphatics & Aromatics Hydrocarbon Ranges**

Analyte	Reporting Limit (µg/m <sup>3</sup> )	Acceptance Criteria	
		Accuracy Limits (%R)	Precision Limits (RPD)
C <sub>5</sub> -C <sub>8</sub> Aliphatics	12	70 - 130	± 25
C <sub>9</sub> -C <sub>12</sub> Aliphatics	12	70 - 130	± 25
C <sub>9</sub> -C <sub>10</sub> Aromatics	10	70 - 130	± 25

**Table 6.15.4 Internal Standards**

Analyte	Accuracy Limits (%)
Bromochloromethane	50 to 200
1,4-Difluorobenzene	50 to 200
Chlorobenzene-d <sub>5</sub>	50 to 200

**Table 6.15.5 Surrogates**

Analyte	Accuracy Limits (%R)
1,2-Dichloroethane-d <sub>4</sub>	70 - 130
Toluene-d <sub>8</sub>	70 - 130
4-Bromofluorobenzene	70 - 130

**Table 16.15.6 Summary of Calibration and QC Procedures**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours.	Compendium of Methods for Toxic Organic Air Pollutants, Method TO-14A, January 1999.	Correct problem then repeat tune.
5 Point Calibration	Prior to sample Analysis.	%RSD ≤30% for APH Target Analyte or hydrocarbon range. Naphthalene is ≤40%.	Correct problem then repeat initial calibration curve.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
LCS (Subset of Target Compounds)	After each initial calibration curve, daily prior to sample analysis.	Recoveries for the APH target compounds and hydrocarbon ranges must be $\pm 30\%$ . If recovery of any compound is above 130%. Analyze samples as long as compound is not detected.	Check the system and re-analyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the beginning of each day.	$\%D \leq 30\%$ for APH target compounds and hydrocarbon ranges. One compound is allowed to be out as long as it is $\leq 50\%D$ . Compound list naphthalene allowed $\%D \leq 40\%$ . If recovery of any compound is above 150%. Instrument must be re-calibrated.	Perform maintenance and repeat test. If the CCV still fails, perform maintenance and a new 5-7 point calibration curve.
Laboratory Blank	After the CCV/LCS.	Results less than the laboratory RL (Tables A-1 & A-2). Naphthalene and C12 are allowed to be 2X the RL.	Inspect the system and re-analyze the blank.
Internal Standard (IS)	As each standard, Blank, and sample is being loaded.	Retention time (RT) for the blanks and samples must be within $\pm 0.33$ min of the RT in the CCV.  The IS area must be within -50 to 200% of the CCV's IS area for the blanks and samples.	<b>For blanks:</b> inspect the system and re-analyze the blank; <b>For samples:</b> If there is not obvious interference with the internal standard, re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. Dilution of the sample to get IS areas within limits may be used if the RL is being obtained.

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Surrogates	As each standard, blank, and sample is being loaded.	70 – 130% R.	<p><b>For blanks:</b> inspect the system and re-analyze the blank;</p> <p><b>For samples:</b> re-analyze sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the 2<sup>nd</sup> analysis. If %R is out-of-limits a 2<sup>nd</sup> time, report data from 1<sup>st</sup> analysis and narrate.</p>
Laboratory Duplicates	Must be 10% of project samples. Duplicate One compound per batch	RPD $\leq$ 30% for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found with the system, narrate.

## **7.0 DATA COLLECTION, REVIEW, REPORTING, AND RECORDS**

### **7.1 DATA COLLECTION**

All analytical results are generated from the instrument software. Data is acquired using a PC/Windows based platform. Data processing occurs on a UNIX based network system. Desktop PCs configured with HP Chemstation software acquire the sample analysis results. Once the acquisition is complete, a post-run macro automatically transfers the raw data files from the hard drive of the acquisition PC to the UNIX server. The UNIX server is a HP D390 server with a full RAID system. This fault tolerant server is configured to manage hot-swappable hard drives and memory cards to avoid any serious downtime. This server is configured with HP-UX 10.2, Thermo Lab Systems Target Software, and Omniback software.

All sample data is stored and processed on the UNIX server. Access to this server is limited based on the privileges associated with the users' passwords. Only the Systems Administrator and the IT Manager maintain full access to the system (which includes exclusive privilege for the adjustment of acquisition station clock times). The system servers are physically located in a secured office, which is locked during off-hours. The data stored on the UNIX server is backed up nightly, weekly, and monthly using a modified grandfather-father-son (GFS) backup rotation. All permanent backup tapes are stored in a secure fireproof safe.

Data reduction of analytical files is accomplished using Thermo Lab Systems' Target software, which allows for complete traceability of the data results. Additionally, multiple permanent records of the data reduction files are maintained through the data back up procedures, minimizing the threat of any lost data trail evidence. Chemists must login to the data reduction software using a

unique password in order to access and work with the sample data files. Once the bench chemists have successfully logged-in to the working environment, all of their activities are tracked and logged by the Target software's electronic assessment trail. The assessment trail file is a tamper proof record of each event that occurred with the data file. The assessment history for a data file contains:

- Date of Change
- Time of Change
- Name of User who made the Change
- Parameter Changed
- Old Value
- New Value
- Reason for Change (if applicable)

The assessment trail file is completely secure within the Target software and cannot be modified or deleted by any user. A hardcopy of the sample assessment trail can be provided upon request for specialized data validation packages. Whenever an electronic raw data assessment is requested, the assessment trail file is automatically included.

Once data reduction is complete, the Scientist or Analyst transfers a copy of the sample results file along with all associated batch QC results into the laboratory's SQL database from which reports are ultimately generated.

### **7.2 DATA REVIEW**

Following analysis, the bench chemist verifies that the computer generated data reduction is correct using the Data Review Checklist (Exhibit 7.2). There are five categories of data review performed in the laboratory.

These categories include:

- I Analytical review performed by the bench reporting chemist. This review includes a review of raw data, verification of all method and project specific QC

requirements, the addition of data qualifier flags when needed, and documentation of any unusual circumstances.

II. Technical review performed by Department Manager or QA-approved peer e.g., analysts who have demonstrated proficiency. This is the same type of review performed in Category I, however, it may be performed either by the same person that performed the analysis or by a second individual if specified by the project profile.

III. QA review performed by a quality assurance specialist. This review is similar to that performed in Categories I and II, however is done with an emphasis on overall quality of the data and verification that standard quality assurance systems are functioning. Data integrity surveillance checks are performed at this level.

IV. Management review by a Director, Department Manager or approved peer. This is a review to ensure the accuracy of the final hardcopy or electronic report. Data integrity surveillance checks are performed at this level.

V. Electronic deliverable review. This review is performed when electronic data deliverables are requested. This review ensures the accuracy of the final electronic report.

Regardless of the TAT, categories I, II, and IV (or I, II, IV and V if electronic reporting is requested) are performed on every data package. As noted earlier analysts who have demonstrated proficiency may perform a category II review. Clients requesting 100% QA review of their data packages receive category III review. Some clients request that 100% of their final data packages undergo a Technical peer review. The review in this case is performed by the Department Manager,

QA-approved peer, or QA personnel. Technical peer review (Category II), must be performed by a different individual than the original analyst, even when that person has the classification of scientist or higher. A request for Technical peer review shall be documented in the project profile.

## **7.3 FINAL REPORT PRODUCTION**

### **7.3.1 AUTOMATIC DATA TRANSFER (ADT) SYSTEM**

Most data reports are created using ADT from the analytical instrument to a custom-reporting module. Approved Analysts/Scientists on each team review the raw data at the instrument and then transfer a copy of the sample results file electronically through a network server to the main database. Once in the database, the data results are automatically formatted into pre-designed method templates using the reporting module. The method templates are designed at sample login and a review copy is e-mailed for client approval prior to reporting. Analysts/Scientists on each analytical team, batch samples results with QC results and any additional information for any sample duplicates or re-analysis.

### **7.3.2 Manual Data Entry System**

Results that cannot be reported using the ADT system are manually entered into a validated, pre-programmed EXCEL spreadsheet. The final report is thoroughly reviewed by an approved team member.

### **7.3.3 Report Compilation**

Data reports are designed to include all necessary information which would be required for traceability including:

- Analytical laboratory name, address, and phone number
- Name and address of the client
- Project name or number (title)

- Total number of pages
- Sample field I.D. number
- Laboratory I.D. number
- Receipt pressure
- Dates of collection and receipt
- Date of extraction (if applicable)
- Date and time of analysis
- Applicable method reference
- Instrument number
- Analytical run file name
- Analyte list
- Dilution factor
- Reporting Limit
- Amount detected in units specified
- Surrogate percent recovery
- Laboratory Director signature
- Chain-of-Custody Record

Each report contains a comprehensive Laboratory Narrative which describes the number of samples received in that batch, any abnormal receipt conditions, any deviations from method specific hold times, the analytical method used, any modifications taken by the lab to the referenced method, and any deviations from standard protocol experienced during sample receiving and analysis. Expected and unexpected deviations that may occur during the analysis of the samples are contained in template format. The Narrative is unambiguous and clearly defines both the nature and substance of the variation.

The QA Manager is responsible for creating, and publishing the templates on a secured and shared network drive. The laboratory staff copies appropriate portions of the template into the Laboratory Narrative document. This approach standardizes the language used in the narratives. The narrative is reviewed using the check sheet in Exhibit 7.2.

The final report is compiled in such a fashion that each subsection is unambiguous and inseparable from the body of the report. A

unique page number appears on every page of the report. The estimated uncertainty of the test results may be included on the report at client request (see Section 8.4).

After all QC results have been reviewed and any deviations from the acceptance criteria are noted in the Laboratory Narrative section of the report, the Laboratory Director, Technical Director, Department Managers or Scientists who are approved by the QA Department for relevant analytical procedures may apply an electronic signature to the final reports. The electronic signature on the report cover page means that the signatory accepts responsibility for the accuracy and completeness of the data generated. The approved signatory corresponds to the Chief Executive Officer/Laboratory Director. The QA Manager keeps a log of the approved applicators of electronic signature to final reports, and ensures that each applicator has the necessary education and experience.

Application of the electronic signature will automatically lock the Work Order thus preventing changes to the original report. If amendments are required due to omissions, errors or additional requests a Work Order reissue is initiated. All reissues receive a unique Work Order number to distinguish them from the original issue. Reissued reports require a reason for the reissue and date of the reissue in the Laboratory Narrative. The laboratory maintains all supporting documentation for the revision including corrections, additions, or deletions relative to the original report.

#### **7.4 ELECTRONIC REPORTING AND REVIEW**

ATL standard format Electronic Diskette Deliverables (EDD) are automatically created in the ATLAS database and sent with the final report. The Lumen EDD software allows the user to create more complex custom/client-specific EDD formats. The Lumen EDD software uses the data from the SQL database for these deliverables, while allowing the users to add custom fields when necessary. The laboratory can produce ERPIMS, GIS/Key, GeoTracker, and EQUIS deliverables. The ATL standard EDD format is delivered in Excel (.xls) format. Other client-specific formats can also be generated. Air Toxics' standard EDD fields are summarized in Table 7.1.

#### **7.5 ECVP/EDD AND REPORTING IN ADOBE FORMAT OR DISKETTE**

eCVP refers to the electronic conversion of laboratory data compiled as a Comprehensive Validation Package in Adobe Acrobat Portable Document Format (.pdf) and archival onto a network drive. The eCVP simultaneously meets the requirements of a Level III or IV Data Validation Package Adobe Corporation's .pdf documents are an exact replica of the original document, but are smaller in file size than the original document format, thereby reducing the amount of storage space required. Adobe Acrobat .pdf provides a convenient way to view and print images at high resolution. The .pdf document is then archived on a network drive and uploaded to a secure Web portal where the client will have access to it using a private username and password provided by ATL.

**Table 7.1. ATL DISKETTE DELIVERABLE STANDARD FORMAT**

FIELD NAMES	FORMAT	WIDTH
LABSAMPLEID	CHAR	15
LABCODE	CHAR	3
MATRIX	CHAR	3
METHOD	CHAR	10
CLIENTSAMPID	CHAR	15
SAMPDATE	DATE	8
ANALDATE	DATE	8
ANALTIME	TIME	4
LABCTLID	CHAR	8
DILUTION	NUMBER	5
REPLMT	NUMBER	5
UNITS	CHAR	4
RESULTS	NUMBER	5
DATAFLAGS	CHAR	2
REPLMT (uG/m3)	NUMBER	5
UNITS (uG/m3)	CHAR	4
RESULTS (uG/m3)	NUMBER	5
DATAFLAGS (uG/m3)	CHAR	2
COMPOUND NAME	CHAR	40
CAS#	CHAR	12
COMMENTS	CHAR	50

LABSAMPLEID:	Sample identifier assigned by ATL.
LABCODE:	Laboratory identifier (ATL).
MATRIX:	Sample Matrix.
METHOD:	Analytical method of analysis.
CLIENTSAMPID:	Sample identifier from Chain of Custody.
SAMPDATE:	The date the sample was collected.
ANALDATE:	The date the sample was analyzed.
ANALTIME:	The time the sample was analyzed.
LABCTLID:	Laboratory batch number.
DILUTION:	Dilution factor.
REPLMT:	Detection limit for sample.
UNITS:	Reporting units of measure.
RESULTS:	Parameter value or result.
DATAFLAGS:	Data qualifiers.
COMPOUND NAME:	The name of each compound analyzed.
CAS#:	The CAS registry number for each compound.
COMMENTS:	General comments field.

**Exhibit 7.1. Example eCVP Cover Page**



AN ENVIRONMENTAL ANALYTICAL LABORATORY

**COMPREHENSIVE VALIDATION PACKAGE**

Modified TO-15

**INVENTORY SHEET**

Work Order #: 0807611

	Page Nos.	
	From	To
1. Work Order Cover Page & Laboratory Narrative	1	5
a. <u>Lumen Validation Report</u>	--	--
2. Sample Results and Raw Data (Organized by Sample)	6	129
a. ATL Sample Results Form		
b. Target Compound Raw Data		
-Internal Standard Area and Retention Time Summary		
-Surrogate Recovery Summary (If Applicable)		
-Chromatogram(s) and Ion Profiles (If Applicable)		
3. QC Results and Raw Data		
a. Method Blank (Results+ Raw Data)	130	136
b. Surrogate Recover Summary Form (If Applicable)	137	137
c. Internal Standard Summary Form (If Applicable)	138	138
d. Duplicate Results Summary Sheet	139	139
e. Matrix Spike/Matrix Spike Duplicate (Results + Raw Data)	--	--
f. Initial Calibration Data (Summary Sheet + Raw Data)	140	268
g. MDL Study (If Applicable)	--	--
h. Continuing Calibration Verification Data (Summary Sheet)	269	282
i. Second Source LCS(Summary + Raw Data)	283	296
j. Extraction Logs	--	--
k. Instrument Run Logs/Software Verification	297	298
l. GC/MS Tune (Results + Raw Data)	299	313
4. Shipping/Receiving Documents		
a. Login Receipt Summary Sheet	314	315
b. Chain-of-Custody Records	316	317
c. Sample Log-In Sheet	318	319
d. Misc Shipping/Receiving Records (list of individual records)		
<u>Sample Receipt Discrepancy Report</u>	--	--
5. Other Records (describe or list)		
a. <u>Manual Spectral Defense</u>	--	--
b. <u>Manual Integrations</u>	--	--
c. <u>Manual Calculations</u>	--	--
d. <u>Canister Dilution Factors</u>	320	322
e. <u>Laboratory Corrective Action Request</u>	--	--
f. <u>CAS Number Reference</u>	323	323
g. <u>Variance Table</u>	--	--
h. <u>Canister Certification</u>	--	--
i. <u>Data Review Check Sheet</u>	324	324

Comments:

Completed by:

**Kara McKiernan**

(Signature)

Kara McKiernan / Document Control

( Print Name & Title)

8/20/08

(Date)

## 7.6 RECORDS OF METHOD CAPABILITY

Prior to sample analysis, the laboratory must demonstrate the ability to meet method accuracy and precision objectives. This is accomplished through an initial multi-point calibration, analysis of four consecutive second source check standards, and completion of a Limit of Detection (MDL) study. The mean recovery for each target analyte must be within current laboratory generated control limits with regard to test and compound. Following this initial set-up, there is a continuing requirement for the demonstration of method capability any time the equipment undergoes significant change, such as different column phase and different concentrator design. Records of these tests are kept for a period of at least 5 years.

There is also a requirement for personnel involved with sample analysis to demonstrate capability for both initial and continuing method proficiency in the specific test method. Analyst/Scientist Demonstration of Capability for the initial method proficiency is accomplished by analyzing any of the following:

- Analysis of four replicate second source check standards either on the same day or on four separate days.
- Successful completion of an independent PT sample.

Demonstration of method proficiency must occur at least once per year to be considered current. Personnel whose demonstration of method proficiency has surpassed the one year criteria may proceed with sample analysis however; their work must be reviewed by the Department Manager or a designated peer until they have completed the continuing method proficiency and received approval by the QA Department. Analyst/Scientist Demonstration of Capability for the continuing method proficiency is

accomplished by analyzing any of the following:

- Analysis of four consecutive LCS either on the same day or on four separate days.
- If four LCS analyses is not possible due to the nature of the work assignment schedule, such as working second shift, a duplicate analysis paired with another Analyst's or Scientist's results that demonstrate acceptable %RPD will be acceptable.
- Successful completion of an independent PT sample.

The Demonstration of Capability for the initial and continuing method proficiency is considered acceptable if the accuracy and precision objectives of the test method are met. Documentation of method proficiency including the relevant raw data summary is kept in each analyst's/scientist's training record. Documentation must be kept for a period of 5 years. The 'Demonstration of Capability Certification Statement' is completed each time a demonstration of method proficiency study is completed.

## 7.7 RECORD STORAGE

The laboratory has a system for record storage such that historical reconstruction of all activities can be made. Raw data includes:

- Instrument run logs
- Instrument calibrations
- Data acquisition files
- Assessment trails
- Manual and spreadsheet calculations
- Date of analysis
- Instrument used
- Sample chain of custody
- Analyst initials and date
- Data review checklists
- Corrective action reports

The laboratory also maintains files dealing with client correspondence. The Client Contacts database stores the date and time of the contact along with a brief summary of the conversation and any decisions made affecting sample status. When a decision is made to proceed with analysis of compromised samples, the contact is logged into the database and a note is made on the Sample Discrepancy Report. The electronic files are maintained for a period of at least 5 years. Additional project management information stored includes:

- The Project Profile
- Client contact database
- Correspondence relating to sample disposition
- Contracts
- SOWs and/or QAPPs

The laboratory maintains electronic and hardcopy reports, as well as supporting information including calibrations, Limit of Detection (MDL) studies, logbooks, and SOPs for a minimum of 5 years. Records stored on electronic media are supported by both hardware and software necessary for retrieval. If the laboratory changes ownership, then responsibility for file storage transfers to the new entity. If the laboratory were to close its doors entirely, then allowance would be made to return files to those clients who contact the laboratory within 30 days of when notice is given. Under either scenario, the transfer of ownership notice would be provided to clients through the NELAP national database and on the ATL web page.

The record keeping system allows for historical reconstruction of all laboratory activities from sample receipt to reporting. The record system includes:

- The identity of personnel involved in sample receiving, preparation, calibration, and analysis.

- A log of names, initials, and signatures for individuals who are responsible for signing or initialing any laboratory record.
- A unique identifier for each piece of equipment used.
- Initials and date for responsible staff at each step in the analytical process.
- Direct, prompt, and legible manual recording in bound logs using permanent black ink.
- Entries in logs that are not obliterated by erasures, over-writing, or markings. All corrections are made by single line strike out of the error followed by the correct entry if applicable. Each strike out is initialed and dated by the person making the correction and the reason for the correction is documented for corrections due to reasons other than transcription errors. Any items, such as computer generated logs or spreadsheets that are pasted into the bound logbook, have initials and date appearing across the item boundary in such a fashion that removal is apparent.
- Records generated by a computer have either hardcopy or write protected back-up copy.
- The QA Department creates and tracks all logbooks throughout their lifetime. Each logbook contains a new logbook request form which is filled out by laboratory personnel and submitted to the QA Department. QA personnel create the logbook by first entering the new logbook into the Inventory Database. This database contains information such as the Book #, its Title, the person's initials and date that created the logbook, the start date, the date it was finished being used and the location of the logbook. Once the necessary information is entered into the database, the logbook is created and given

to either the Department Manager or the person that submitted the request. When the logbook is completed or no longer in use, it is submitted to the QA Department. QA personnel update the Inventory Database with the finished date, the location and the logbook is archived.

## **7.8 CONFIDENTIALITY OF DATA**

The data generated by analyzing a sample is considered to be the property of the entity appearing in the “**BILL TO:**” field of the Work Order request unless other contractual arrangements have been made. Accordingly, that data is treated as confidential information and released only to that client, as identified by associated contractual agreements unless written permission is given to proceed otherwise. All data generated under NELAP related fields of testing shall be made available to recognized agents of any laboratory accrediting authority for purposes of inspection and verification during an onsite visit. Clients will not be notified when the accrediting authority reviews data during the normal course of the onsite assessment. Clients will be notified any time a request is made by the accrediting authority to remove

copies of sample files, either electronic or hardcopy, from the laboratory. Client written approval must be arranged prior to removal of the files from the laboratory unless the request is accompanied by appropriate court order. Both e-mail and facsimile data are treated as confidential by noting on the cover page:

*“The information contained in this communication is confidential and intended only for the use of the individual or entity named above. Any other use, dissemination, distribution, or copying of this communication is prohibited. If you have received this communication in error, please notify us by telephone and return the original message to us via US mail.”*

Client confidentiality is observed in accordance with guidelines described in NELAC Chapter 5.5.10.6 (2003):

*“The laboratory shall ensure that, where clients require transmission of test results by telephone, telex, facsimile or other electronic or electromagnetic means, staff will follow documented procedures that ensure the requirements of this Standard are met and that confidentiality is preserved.”*

Exhibit 7.2.

@Air Toxics Ltd.

**DATA REVIEW CHECKLIST**      Work Order #:

A <sub>1</sub>	A <sub>2</sub>	R	T	M	Q	
<input type="checkbox"/>	Analysis/Reporting vs. Project Profile/SOP requirements checked (i.e. 100% Dups, J-Flag to MDL, etc)					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	The final report has the correct reporting list, special units, and header info.
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Lab Narrative is correct (proper method & description/Receiving & Analytical notes correct)
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Sample Discrepancy Report (SDR) is completed
<input type="checkbox"/>	Corrective Action issued - # _____					
<input type="checkbox"/>	Unusual circumstances have been documented in the notes section below					

**LUMEN validation report present and initialed**      **CIRCLE (YES / NO)**

<input type="checkbox"/>	Lab Blank, CCV, LCS and DUP met QC criteria					
<input type="checkbox"/>	Hold time is met for all samples					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Appropriate data qualifier flags are applied
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Manual integrations for samples and QC are properly documented
<input type="checkbox"/>	Samples analyzed within the project or method specific clock					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Retention times have been verified
<input type="checkbox"/>	Appropriate ICAL(s) included					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	At least one result per sample is verified against the target quant sheets/raw data
<input type="checkbox"/>	Dilution factor correctly calculated (sample load volume, syringe and bag dilutions, can pressurization(s))					
<input type="checkbox"/>	Correct amount of sample analyzed (i.e. sample not over-diluted)					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Spectra verified - documentation of spectral defense included (Section 5A of eCVP pkg)
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	TICs resemble reference spectra
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	TICs between duplicate samples are consistent
<input type="checkbox"/>	Checked samples for trends (i.e. Influent vs. Effluent, Field Dups, Field/Trip Blank, etc.)					
<input type="checkbox"/>	Data for multiple analyses of sample(s) has been evaluated for comparability of results					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Special units for all samples in the final report are correctly calculated
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Manually entered results checked (i.e. TPH/NMOC)
<input type="checkbox"/>	Chain of Custody verified for any special comments (i.e. different compounds/RLs, action levels)					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Chain of Custody scanned correctly
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Verify sample id's vs. chain of custody
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Date MDL(s) performed per instrument(s)
<input type="checkbox"/>	Samples pressurized w/ appropriate gas (N <sub>2</sub> or He) <input type="checkbox"/> Other (i.e. Tedlar bag, cartridge, sorbent)					
<input type="checkbox"/>	Final pressure consistent with canister size (6L vs. 1L)					
<input type="checkbox"/>	Verify receipt pressures					
<input type="checkbox"/>	Verify canister ID #'s					
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	Final invoice amount correct (adjusted for TAT, Penalties, Re-issue Charges etc.)
		<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/>	MDL date(s) present for all instruments utilized
	<input type="checkbox"/>	Client LUMEN report reviewed for accuracy and completeness				

Notes: (to include: noting samples with QA/QC problems, Blanks with positive hits, narratives, etc.)

A/R:

M/O:

A <sub>1</sub> /A <sub>2</sub>	R/T	M	Q
(Analytical Review/Date)	(Reporting Review/Date)	(Management Review/Date)	(QA Review/Date)
A <sub>1</sub> :	R:		
A <sub>2</sub> :	T:		

Note (1): Please check all the appropriate boxes. Indicate "NA" for any statement that does not apply.      Rev. 02/20/09  
 Note (2): Management reviewer and reporting reviewer must be separate individuals.

## **8.0 ESTABLISHING ACCEPTANCE CRITERIA**

### **8.1 CONTROL CHART PROGRAM**

Air Toxics Ltd. complies with guidance from ISO/IEC 17025:2005(E), Section 5.9, to determine quality control limits. This regulation suggests that statistical techniques may be used to detect trends, but does not mandate acceptance or rejection of analytical results based on use of historically derived control limits. Additionally, NELAP does not address the issue of control charting. Therefore, in accordance with ISO/IEC 17025:1999(E), Section 5.9, quality controls are in place to monitor validity of tests and calibrations only.

Historically derived control limits are generated twice annually by the QA Department, or whenever a procedure has been changed significantly. Control Limits may be updated less often (or not at all) for methods which are performed so infrequently that it is difficult or impossible to gather at least 20 data points. These limits, however, are not used to validate data unless required by specific client request. A complete description of the Control Chart program can be found in ATL's SOP #48.

### **8.2 ESTABLISHING CONTROL LIMITS**

Control limits are generated from a minimum of 20 randomly chosen data points. The calculations used to establish and update these investigative limits include:

Upper Control Limit	= M	+3S
Lower Control Limit	= M	-3S
Upper Warning Limit	= M	+2S
Lower Warning Limit	= M	-2S

Where:

M: The population mean recovery of at least 20 points, and

S: The standard deviation of the population.

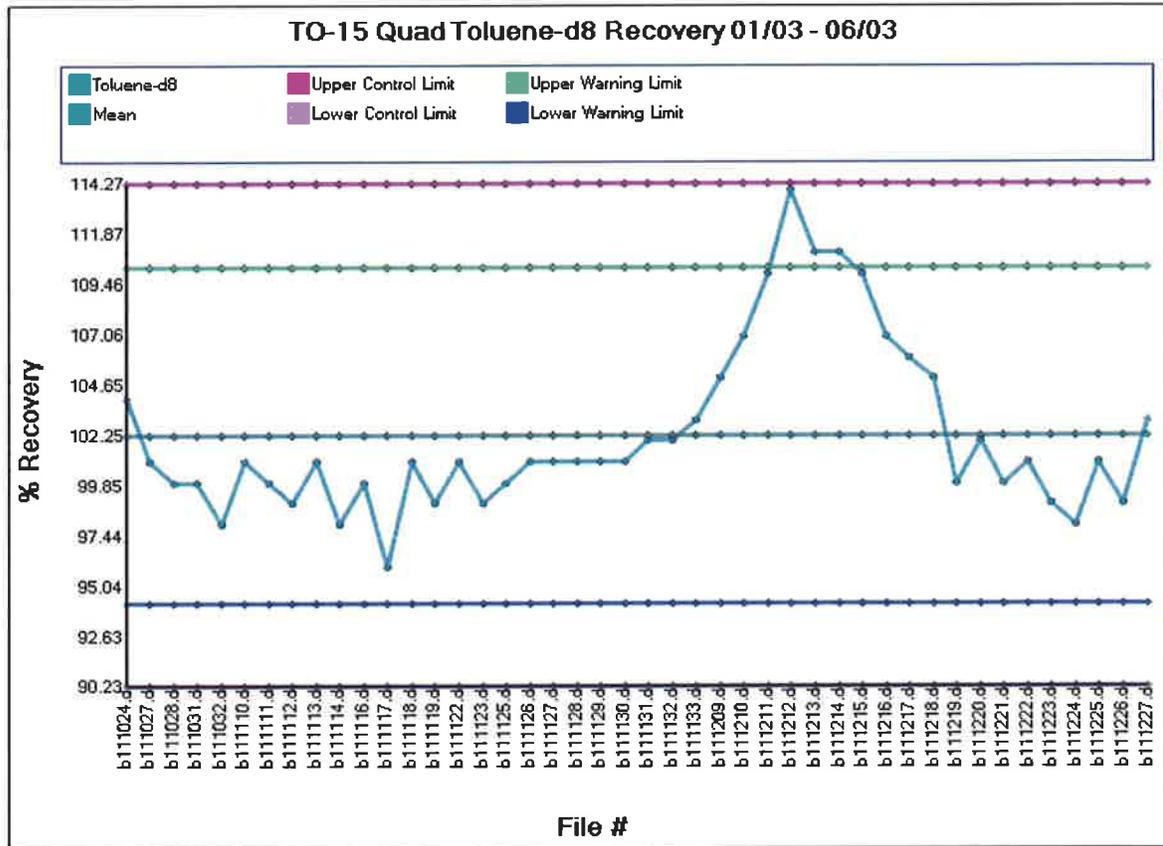
### **8.3 INTERPRETING CONTROL LIMITS**

Calculated control limits based on historical data for Surrogate and LCS recoveries are used to demonstrate statistical control and display method variability, and are not used to qualify actual sample recoveries. Additionally, control limits may not be representative of the analytical process if less than 20 points are generated for a given method. As a result, Air Toxics Ltd. uses the default limits prescribed for each method in the corresponding SOP and Section 6.0 of this document. Historically derived control limits are used to evaluate LCS or Surrogate results only when requested by clients or certifying agencies.

### **8.4 MEASUREMENT UNCERTAINTY**

Upon request from a client, Air Toxics Limited will report measurement uncertainty for a given analyte reported by a specific method. Measurement uncertainty is calculated as a function of historical LCS Control Limits (suggested by the American Association for Laboratory Accreditation Guide for Estimation of Measurement Uncertainty in Testing, July 2002). This policy is valid for environmental test methods in which measurement uncertainty is not defined. Uncertainty is estimated using standard deviation of Laboratory Control Samples of more than 20 points. Measurement uncertainty is estimated to the 95% confidence interval and expressed as  $\pm 2X$  the standard deviation of the mean percent recovery of each given analyte. If 20 points do not exist from the current control limit calculation, the collection period may be expanded.

Exhibit 8.1. Control Chart



## **9.0 PREVENTIVE MAINTENANCE**

The best form of preventive maintenance is to have good, new stuff and a lot of it. If the customer expectations for quality, turn around time, and price are to be met, then the instrumentation must be maintained in a fashion that supports the quality objective. The program is designed to adequately protect the laboratory from unexpected instrument failure and minimize scheduled instrument down time. Preventive maintenance consists of an on-going program of routine maintenance, service contracts, and a comprehensive inventory of spare parts.

The likelihood of unscheduled down time tends to increase as instrumentation ages. A three year lease term provides nearly optimum instrument life cycles. At the end of the lease term, the equipment will be exchanged for new models.

### **9.1 ROUTINE MAINTENANCE**

The Analyst/Scientist monitors instruments for potential failure on a daily basis. The analysis of blanks and control standards at the start of the day and as analysis continues helps to provide real time feedback to the Analyst/Scientist on the condition of the instruments. Routine maintenance, specific to the various types of instruments, is covered in the method SOPs.

Any routine or major maintenance is documented in the bound maintenance logbook assigned to each instrument. The date of the maintenance, work performed, and Analyst's or Scientist's initials are included.

If a malfunction occurs and control of the analytical system cannot be demonstrated using the QC parameters, discussed in section 4.3, the instrument is removed from production until analytical control can again be demonstrated.

## **9.2 SERVICE CONTRACTS**

Some analytical systems are covered under manufacturer service agreements. These agreements cover all forms of hardware failure and include regular hardware upgrades as needed. The response time is guaranteed to be within 48 hours under the agreement and includes parts and labor.

Some contracts cover regularly scheduled routine maintenance. Leased instrumentation is similarly covered by service agreements either through the leasing agency or directly with the manufacturer.

In addition, the Technical Services group performs bi-annual (every six months) preventative maintenance on the mass spectrometers. These records are kept in the individual instrument's maintenance logbooks.

### **9.3 SPARE PARTS INVENTORY**

A normal inventory of analytical consumable parts most frequently required is maintained in the laboratory. These parts are typically not covered by the service agreements and may take several weeks to acquire on an as needed basis. An inventory is required to minimize instrument down time and facilitate routine maintenance. An inventory of design parts is also maintained including:

- Stainless steel valves
- Tubing
- Various connecting nuts and ferrules
- Tools
- Flow controllers
- Flow sensors
- Electrical connectors
- Sheet metal
- Abundance of miscellaneous items
- Multipliers and other MS source parts

The laboratory invests a significant amount of money every year in lab/computer and research supplies.

#### **9.4 CONTROL OF MISCELLANEOUS MONITORING, MEASURING, TESTING, AND DATA COLLECTION EQUIPMENT**

In addition to the equipment used directly in the analysis of samples, ATL uses various other monitoring, measuring, testing, and data collection equipment. This equipment includes: analytical balances and weight sets, pressure gauges, flow meters, fume hood testing devices, thermometers, temperature and humidity recorders, mechanical volumetric devices, oven vacuum gauges, and sampling interface flow controllers. The procedures for ensuring the accuracy of the test equipment are summarized in the following sections. Additional information can be found in ATL Certification of Test Equipment SOP, #34, and Refrigerator and Freezer Temperature Monitoring SOP #19.

##### **9.4.1 Analytical Balances and Weight Sets**

The analytical balances are certified and serviced once a year by an independent balance maintenance company. A sticker is put on the side of the balance to indicate the date of certification and the company performing the certification. The certificates are maintained in the Quality Assurance (QA) Department. The certificate must indicate that the reference standards are traceable to NIST standards and indicate the tolerances of the balance.

In addition, each time a balance is used; it is first checked with Class 1 weights. The weights used must bracket the final amount being weighed. The result must be within acceptance criteria. If the acceptance criteria are not met, a Corrective Action Request (CAR) form is initiated and given to the QA Department. The balance and/or weight set may require servicing to correct the problem.

Annually, all Class 1 weight sets are serviced and calibrated on-site against NIST-certified

standards by an independent calibration company. The certificate of calibration is maintained in the QA Department. The weights are kept in the manufacturer package that indicates the certification expiration date for the weight set. A sticker is put on the outside of the box to indicate the date of certification and the company performing the certification.

##### **9.4.2 Pressure Gauges**

Pressure gauges are used to verify sample receipt pressures and for gaseous standard preparation. The measurement of pressure on the gauges used to pressurize canisters is relative. The readings are used to assess the initial canister receipt vacuum/pressure and then pressurize the canister to a known pressure. The pressurization Technician compares the final vacuum/pressure recorded on the Chain-of-Custody Record and/or sample tags by the field personnel with the receipt vacuum/pressure. If there is a discrepancy of more than 7"Hg/7psi a Sample Discrepancy Report is initiated and the client is notified.

In addition, the pressure gauges installed on the pressurization manifolds are re-calibrated and NIST certified on-site by an independent calibration company annually or as-needed. The certificates are kept on file in the QA Department.

##### **9.4.3 Fume Hood Testing Device**

Quarterly, the Velocichck Portable Air Velocity Meter is used by a member of the Safety Committee to check fume hood velocities. Velocities are checked in various quadrants of the hood at both full open and half-open sash levels. Results of this check must be within specified limits and are recorded in the Fume Hood Evaluation Logbook. If results are outside of these limits, the fume hood must be taken out of service until the problem is corrected.

Annually, the Velocichек Portable Air Velocity Meter is calibrated on-site by an independent calibration company against NIST-traceable standards. The certificates of calibration are maintained in the QA Department.

#### **9.4.4 Thermometers**

##### **9.4.4.1 Reference Thermometers**

ATL has NIST-traceable digital thermometers, which are used by the QA Department as reference devices. The thermometers are re-calibrated and certified annually by an independent calibration company. A label indicating the date of calibration, the due date for the next calibration, and the name of the company performing the certification is placed on the back of the thermometer itself. The reference thermometers are kept, along with certificates of calibration, in the QA Department.

##### **9.4.4.2 Working Liquid-Filled (Ref/Freezer and Receiving) Thermometers**

Thermometers used to record the temperature of refrigerator/freezers as well as of Temperature Blanks (received with samples shipped on ice), are re-certified every year by the QA Department using the NIST-traceable digital thermometer as reference. The certification test is performed by comparison to the NIST-traceable digital thermometer. Both thermometers (working and reference) are placed in a Dual Well Dry Block Calibrator (Model 9009 Hart Scientific). This instrument allows accuracy checks at both low and high temperatures. Alternatively, the certification test may be performed by placing the thermometers in a medium to large-sized beaker filled with water (or Methanol for freezer thermometers). The beakers are placed into a refrigerator (or freezer) for approximately one hour. The beakers are then removed from the refrigerator/freezer and the thermometer readings are compared to the

NIST-traceable digital thermometer which is also submersed in the liquid.

The temperature range tested must correspond to the temperature range that the thermometer is used to measure (i.e. approximately  $4 \pm 2^{\circ}\text{C}$  for Refrigerator and Receiving thermometers and  $\leq -10^{\circ}\text{C} \pm 5^{\circ}\text{C}$  for Freezer thermometers). The results of this test are recorded in the Thermometer Calibration Verification logbook. The difference between the temperatures of the working thermometers and the reference thermometer should be within the specified accuracy limits. Any thermometer that fails this certification test is discarded and new replacements are purchased as needed.

The manufacturer provides the newly purchased thermometers with a calibration certificate. The QA Department checks the new thermometers for defects (i.e., air bubbles present in red liquid column) before placing the new thermometers into use. A table containing the exact location, serial numbers, calibration and re-calibration due dates of each thermometer is kept in the Thermometer Calibration Verification Logbook.

##### **9.4.4.3 Oven and IS Station Thermometers**

Temperature controllers used in the Canister Cleaning and Tube Preparation areas are verified on a yearly basis by the QA Department to ensure that the proper temperature range is being achieved. The test is performed using the NIST-traceable digital thermometer as a reference. The test consists of comparing the temperatures displayed by the ovens temperature controllers versus the temperature measured by the reference thermometer.

The temperature range tested must correspond to the temperature range the thermometer is used to measure (i.e. approximately  $65 - 125^{\circ}\text{C}$  for Can Cleaning ovens). The temperature readings are recorded in the Thermometer Calibration Verification logbook, which is

kept in the QA Department. The accuracy limits used to compare the two readings are  $\pm 5^{\circ}\text{C}$ .

If the readings are outside of acceptance limits, a correction factor may be applied to the temperature readings and maintenance or replacement of the controller may be necessary.

Temperature control used for desorption at the Internal Standard (IS) Loading Station in the main lab (used for VOST and TO-17 analysis) is verified for accuracy prior to analysis using a NIST-traceable digital thermometer as a reference.

The temperature range tested must correspond to the temperature range the thermometer is used to measure (i.e. approximately  $100^{\circ}\text{C}$  for the IS station). The temperature readings are recorded in the instrument logbook. The acceptance limits are  $\pm 10^{\circ}\text{C}$ .

If the readings are outside of acceptance limits, a correction factor may be applied to the temperature readings and maintenance or replacement of the thermometer may be necessary.

#### **9.4.4.4 Non-Contact Thermometers**

Non-contact thermometers are used to verify the temperature of the desorption plate used in the analysis of VOST and TO-17 samples as well as to take the temperature of chilled samples which arrive without a Temperature Blank. These thermometers are certified against NIST-traceable standards on-site by an independent calibration company on a yearly basis. Certificates of calibration are maintained in the QA Department.

#### **9.4.5 Temperature/Humidity Recorders**

A Temperature/Humidity Recorder is used to verify that conditions required for PM10/TSP analyses of filters have been met in the Desiccator unit. The required conditions are a

temperature of  $59$  to  $86^{\circ}\text{F} + 5^{\circ}\text{F}$  and humidity at  $20$  to  $45\% \text{RH} \pm 5\% \text{RH}$  ( $\leq 50\% \text{RH} \pm 5\% \text{RH}$  for TSP) over a 24 hour period. These conditions are graphed, and every 7 days a replacement graph card is placed into the recorder by designated personnel. The date range recorded, along with the analyst's initials, is noted on the back of the graph and filed in a folder next to the instrument. The recorder is re-calibrated and certified annually by an independent calibration company. The certificates of calibration are kept on file in the QA Department.

The Refrigerator used for the storage of VOST samples uses a Temperature Recorder in order to verify required temperature has been maintained over holidays when the regular temperature checks are not performed (see ATL SOP #19 Refrigerator/Freezer Temperature Monitoring and Documentation). The temperature is graphed, and every 7 days a replacement graph card is placed into the recorder by designated personnel. The date range recorded, along with the analyst's initials, is noted on the back of the graph and filed in a folder next to the instrument. The recorder is re-calibrated and certified annually by an independent calibration company. The certificates of calibration are kept on file in the QA Department.

#### **9.4.6 Flow Meters**

Flow meters are used in the Laboratory to check the flow rates for VOST/TO-17 and other analyses, and in connection with sorbent tube preparation. Canister Cleaning also uses flow meters to calibrate flow controllers. These instruments are re-certified annually on-site by an independent calibration company against NIST-traceable standards. The certificates of calibration are kept in the QA Department.

#### **9.4.7 Mass Flow Controllers**

The Mass Flow Controllers on the sampling interfaces are used as part of the Initial

Calibration. Therefore, measurements made using them are relative in nature. The samples are introduced through the very same process; therefore any potential bias is self-correcting. In addition, the accuracy of the Mass Flow Controllers is verified in four ways:

- 1) Each time the daily CCV is analyzed, the recoveries document the accuracy of the Mass Flow Controller with respect to the most recent instrument Calibration.
- 2) The linearity of the Calibration Curve demonstrates the accuracy of the Mass Flow Controller because the curve is developed using a mixture of syringe and Flow Controller standard loadings.
- 3) The accuracy of the Mass Flow Controller is verified through comparison of the new Calibration Curve with the previous Curve
- 4) In addition, the Mass Flow Controllers on the sampling interfaces are calibrated in house using a NIST certified flow meter before each Initial Calibration. They are calibrated by trained Analysts and/or Scientists. This action is documented in the instrument logbook the day of the calibration which includes flow controller serial number, NIST flow meter expiration date, nominal value, actual value, verified/set by initials and date. The Laboratory staff oversees the Mass Flow Controller certification program. The certificate of calibration for the NIST flow meter is kept in the QA Department.

#### **9.4.8 Mechanical Volumetric Devices**

Mechanical volumetric devices such as solvent dispensers are verified for accuracy against a known volume approximately once per month and never less than four times per year.

#### **9.4.9 Oven Vacuum Gauges**

Each oven used by the Support Services Department to clean stainless steel canisters is equipped with a CONVECTRON vacuum gauge and controller. The accuracy of these gauges is checked approximately every 6 months or as needed.

A NIST certified CONVECTRON gauge and controller is mounted onto an empty port on the evacuation manifold by a member of the Support Services Department. The controller readings are compared to the oven vacuum gauges and recorded into to the comment line of the oven logbook. The NIST gauge and the oven gauge should match within  $\pm 6\%$  ( $\pm 1.2$  mTorr for a 20 mTorr reading) based on the manufacturer's accuracy limits. If the 2 gauges do not match, then the oven vacuum gauge controller is adjusted until the readings are the same, or the oven gauge is replaced/repared.

Documentation of changes or repair is noted in the Canister Cleaning Maintenance Logbook. The NIST gauge is re-certified annually on-site by an independent calibration company and the certificate of calibration is kept in the QA Department.

## **10.0 PROFICIENCY TESTING PROGRAM**

### **10.1 NELAP PT SAMPLE PROGRAM**

Proficiency testing (PT) samples are used to measure analytical accuracy, precision, and report completeness. To be accredited under NELAP, the laboratory contracts with an outside approved PT sample provider in each field of testing. Testing is limited by availability of samples that meet NELAP criteria (noted below). The provider must be a NIST accredited PT provider. It may be necessary to participate in more than one proficiency testing program to be evaluated for multiple interdependent analyte groups. Currently there is no NELAP accredited PT provider for air samples therefore, ATL participates in a PT program for EPA Method TO-15 which is ISO 17025 compliant. Performance samples are processed through the laboratory in the same manner as project samples. In each calendar year, the certified lab will complete at least two separate proficiency testing samples for each analyte or interdependent analyte group. The following policies apply to laboratory PT sample analysis and reporting:

- The samples shall be analyzed and reported to the PT provider within 45 calendar days of receipt or the specific deadline specified by the PT provider.
- The PT sample is received and logged into an electronic sample receiving database in the same fashion as field samples.
- The laboratory must follow the PT provider's instructions for preparing the PT sample.
- The laboratory management and bench chemist ensure that the PT samples are prepared, analyzed and reported in the same fashion as field samples using the same staff, equipment, and methods.
- Initial and continuing calibrations for the PT sample are analyzed at the same frequency of field samples.
- The PT sample is analyzed with the same quality control samples as routine field samples.
- The PT sample cannot undergo duplicate or replicate analyses that would not ordinarily be performed on field samples. The PT sample result cannot be derived from averaging the results of multiple analyses unless specifically called for in the reference method.
- The PT sample can only be analyzed on equipment leased or owned by the company and handled only by bona fide employees of the company.
- The analysis of PT samples by temporary or contract employees is explicitly forbidden.
- The laboratory shall not subcontract any PT sample or portion.
- The laboratory shall not knowingly receive any PT sample or portion from another lab.
- The laboratory shall not communicate in any fashion with another laboratory concerning the PT sample or results.
- The laboratory shall not attempt to obtain the PT sample result prior to reporting.
- The PT sample reporting forms provided by the sample provider will be used to report the results and will be maintained in the laboratory's record system.
- The laboratory shall maintain copies of all written, printed and electronic records relating the analysis or reporting of the PT sample for a period of 5 years or as

required by the applicable regulatory program.

- A CAR form will be generated any time an analyte result fails the proficiency testing assessment. A copy of the PT results is sent to the NELAP accrediting agency and associated corrective action summary will be sent upon request.
- The lab authorizes provider to release any PT assessment information to the accrediting agency.
- The QA Manager must sign the PT results form and by so doing, attests that the sample was analyzed and reported in the same fashion as a field sample and followed the PT provider instructions for preparation.
- The lab must notify its primary accrediting agency and any other agencies under reciprocity that it has enrolled with a particular PT provider.
- The lab must notify its primary accrediting agency and any other agencies under reciprocity in the event it wishes to change PT providers.
- For each analyte or interdependent analyte group for which proficiency is not available, the certified lab will establish, maintain and document the accuracy and reliability of its procedures through a system of internal quality management.
- Results of any failed PT samples are summarized in the Quarterly QA Status Report.

## **10.2 EXTERNAL (NON-NELAP) PT SAMPLES**

Occasionally proficiency testing samples are submitted along with field samples by private clients. The lab processes and reports the

samples in the same fashion as field samples. When the client notifies the laboratory that one or more analytes appear to have failed, the report is processed through the normal Client Inquiry Corrective Action Process. The QA Manager will carry out an assessment and investigation into the circumstances surrounding the proficiency results including aspects relating to how the client prepared the sample for submission. The outcome of the assessment will be documented as per (Section 3.3.2) and maintained on file for a period of 5 years. Results of any failed external PT samples are summarized in the Quarterly QA Status Report.

## **11.0 MANAGEMENT OF COMPUTER AND SOFTWARE SYSTEMS**

Data are electronically captured from virtually all analytical instruments used by Air Toxics Limited. A network of computers and servers is used for the acquisition, processing, manipulation, recording, storage, and retrieval of test data. The laboratory uses a variety of both commercial as well as proprietary software applications to acquire, process, and report sample results. This network of computers is also used to receive and process customer information regarding field activities, sample disposition, and quality assurance objectives. Quality systems relating to the management of the computers and software are designed to incorporate the standards established in the *EPA Document "2185-Good Automated Laboratory Practices (1995)"* wherever possible given the size and resources available in the laboratory and IT groups.

### **11.1 SECURITY**

The systems of Air Toxics Ltd. are protected from unauthorized access through the use of both physical and programmatic security measures. All of the laboratory servers are housed in a locked office, which maintains favorable environmental conditions to allow for optimal server performance. Access to the laboratory's networks is granted by the Systems Administrator or IT Manager. Network access is tightly controlled for the entire company. Users maintain individual network accounts and are allowed to access specific areas of the network based on the privileges assigned to them. A user is granted access to only those areas needed to fulfill his/her job function. All software used to reduce sample data or generate sample reports is password protected; users are granted rights to these systems based on a read/write/none privilege system.

### **11.2 BACK UP AND STORAGE OF DATA**

All data systems are backed up on a daily, weekly, and monthly basis using a modified grandfather-father-son (GFS) rotation protocol. Specifically, these back ups are conducted on the servers responsible for all laboratory production data files and databases (i.e., Project Management files, analytical data, audit trails, quality assurance documents, etc.). A daily incremental back up is scheduled to run each night Monday through Saturday. The daily incremental back up is limited to files modified the same day. On Sunday, a weekly full back up of all files on each server is completed. At the end of each month, a full back up of each data system is conducted. This monthly back up tape is then placed in permanent storage. The permanent historical back-up tapes are stored in a fireproof safe in the secured server office. Data is not removed from the server until at least three permanent monthly back-up tapes have been created. This ensures that no archived data will be lost due to corruption of the magnetic tape. A more comprehensive description of the electronic data archiving system can be found in ATL SOP #55, *Electronic Archival of GC/MS Analytical Instrument Data*.

### **11.3 SOFTWARE AND ELECTRONIC DATA VALIDATION**

The IT Department is responsible for the testing and verification of all internally developed software applications. This includes testing during the software development, testing of the first pre-release version (alpha testing), and testing the release version in a closely monitored production environment. Findings discovered during the alpha and beta tests are documented and software fixes are applied as warranted. All custom software applications are tested prior to their release to the production laboratory.

There are three stages of testing and implementation of custom software modules by the IT Department.

### **11.3.1 Stage I – Alpha Testing**

Stage I testing is conducted for all ATLAS modules developed at Air Toxics Ltd. The Stage I testing is performed by two members of the ATLAS development team, typically the lead programmer and a representative end-user. The lead programmer is responsible for preliminary testing of the application and fixing errors, “bugs,” within the program. The other evaluator acts as the user, testing the functions and features of the ATLAS module. The alpha test performed by the representative end user is conducted at a production workstation, which maintains the typical ATLAS configuration and connects with the ATLAS database through the laboratory network. This Stage I testing is conducted off-line, as the module is not used to perform any real-time production work. During the testing, the alpha tester identifies errors within the program and reports them to the lead programmer by opening an incident in the software program c.Support. Examples include: finding areas where the module does not perform the task as per the specifications or an error message is displayed on the screen when working with the module.

The lead programmer evaluates the errors that are reported in the incident and fixes all identified problems within the ATLAS module, documenting the status of the repair in the incident in the c.Support database (Exhibit 11.1).

Once all main functions have been tested and the alpha tester can no longer identify significant bugs within the program, the alpha testing is concluded. The alpha test results are documented in the completed incident, which are maintained as a permanent record in the c.Support database.

### **11.3.2 Stage II – Beta Testing**

The Stage II testing is conducted after completion of the alpha test. This testing is performed by select on-line end-users. End-user participants in the beta test are chosen by their Department Manager, in an effort to have an accurate representation of the end-users that will be using the ATLAS module. The beta test participants perform their job duties using the ATLAS module. Both the lead programmer and the IT Manager are available during the beta test.

The beta testers are trained how to use the ATLAS module on-line. The lead programmer, IT Manager, and the Department Managers all participate in the training of the beta testers. The beta testers perform their production duties on the ATLAS module and check all output from the module for accuracy. A summary of the beta test may be documented (Exhibit 11.2).

The beta testers identify any errors or deficiencies within the application and notify the lead programmer immediately upon finding a bug. In addition, the beta testers submit an incident via the c.Support database to the programmer.

The lead programmer evaluates the errors that are reported in the incident and fixes all identified problems within the ATLAS module, documenting the status of the repair in the incident in the c.Support database.

The new version of the ATLAS module is uploaded onto the computer for the beta tester. The testing is resumed at the beginning of the task from which errors were identified in the software application. The tester verifies the fixes for reported bugs and documents this verification in the incident.

After the occurrence of bugs has been mitigated, the beta testing is concluded. The beta test results are documented in the

completed incident, which are maintained as a permanent record in the c.Support database.

### **11.3.3 Stage III - Implementation**

Stage III implementation is initiated after completion of the beta test. All end-users are given access to on-line module. Training for end-users is conducted individually or as a group by a member of the ATLAS development team and/or the Department Managers.

If any bug occurs on-line, a member of the IT team is contacted and the problem assessed to determine if it is a bug or a training issue. A confirmed bug is documented as an incident in the c.Support database. Any problem that affects data integrity is documented separately. This type of problem is typically documented by the IT Manager; however other members of the development team can assume this responsibility. This documentation is kept as a permanent record in the ATLAS project documentation binder in the IT Manager's office.

The lead programmer evaluates any error reported in the incident and fixes the program within the ATLAS module, documenting the status of the repair in the incident in the c.Support database. The new version of the ATLAS module is uploaded to all PCs running the application. In most cases, an automatic update program loads the new version of the application for all users when they re-enter the program. Production work continues with the new version of the module.

### **11.3.4 Commercial Software Data Validation**

Any commercial software application used for data reduction is verified for adequate performance before release to the production laboratory. The various testing stages and process for implementation of internally developed software are described in ATL SOP #47, *Implementation and Testing of ATLAS Modules*.

**Exhibit 11.1. ATLAS Incident Report**

Dialog - Print Incident

Page 1 of 1

**Incident**

[Print](#) [Change Options](#) [Close Window](#)

Dates and times are displayed in the (GMT-08:00) Pacific Time (US & Canada) time zone.

**Customer Details**

Name:		Customer ID:	
Location:		Department:	Sales
Company:	Air Toxics	Phone:	
Email:			

**Details**

Number:	98DH39296A	Categorization:	IT
Status:	Open		ATLAS
Priority:	Emergency		

Effective SLA: IT

Opened: 8/13/2009 4:28:18 PM by

Closed:

**Custom Categories**

Issue Type: Bug

**Description**

In Method Editor, trying to create list for TO-11A and get "Run Time Error", then Atlas kicks me out.

**Resolution**

**Exhibit 11.2. ATLAS Beta Testing Summary Form**

Name of Tester: \_\_\_\_\_

Date: \_\_\_\_\_

Module Tested: \_\_\_\_\_

Summary of Beta Test (include brief description of criteria used, etc.):

---

---

---

---

---

---

---

---

---

---

Did the module pass all tests conducted?

Yes

No

If no, please provide comments below:

---

---

---

---

Signature of Tester: \_\_\_\_\_

Module approved for release to production group?

Yes

No

Signature of IT Manager: \_\_\_\_\_

Date: \_\_\_\_\_

**This page left blank intentionally.**

## 12.0 CONTROL OF PURCHASED ITEMS AND EXTERNAL SERVICES

The primary materials procured by the laboratory are computer hardware, analytical software, standard office software, analytical instrumentation, certified standards, carrier gases and cryogenics, miscellaneous laboratory supplies, NIST-traceable re-certifications, disposable sampling media (e.g., Tedlar bags), and service contracts.

Control of the purchase of these items and services is maintained using a standard purchase order system that includes the following:

- A purchase request that is approved by a Director or Manager.
- An assigned purchase order (PO) number that is logged along with the date, vendor, and requester.
- A requirement that upon receipt or delivery of services, the product is inspected by the purchasing agent, and compared to the packing slip and/or request for services.
- Each PO is matched with invoices prior to payment to insure that purchased items or services were delivered as expected.

Critical vendors (those for whom a failure or lack of supply would cause irreparable damage to the laboratory operations) are selected on the basis of either being the sole supplier of an item or as a result of reliable service over a several year period. A table of current critical suppliers is presented in Exhibit 12.1.

When reagents are purchased in bulk volume for laboratory use, each lot is certified for cleanliness prior to use. A laboratory blank is prepared using the analytical reference method. The concentration of target species present must be less than the laboratory reporting limit for the lot to be certified clean

and approved for use. Once a lot is approved, the vendor will set aside stock sufficient for several months for ATL use. The certificates of analysis are kept on file in the main analytical laboratory and each incoming shipment is monitored by the extractions area staff to verify that only certified lot numbers are used in the processes.

<u>Solvent</u>	<u>Certification Method</u>
Methylene Chloride	SW8270C
Hexane	TO-4A
Water	SW8260B/SW8270C

Each reagent is labeled with the date of receipt, date opened, and date of expiration. In the case of Methylene Chloride and Hexane, a 100 mL aliquot of the solvent is evaporated to 1 mL and analyzed via method SW8270C or TO-4A. In the case of reagent water, a reagent blank is prepared and analyzed via methods SW8260B (5 mL) and SW8270C (1 L). Only reagent grade water is used in the laboratory and is purchased in bulk lots as HPLC grade.

Cylinder gasses used as diluent gas for sample analyses are also certified for cleanliness. Each tank of UHP Helium or Nitrogen is tested by filling a clean lab blank canister followed by analysis via EPA method TO-14A/TO-15. All target species must be present at less than the reporting limit in order for the cylinder to be certified clean. Results of the analysis are posted on each cylinder and stored in designated logbooks.

NIST traceable standards are re-certified upon receipt. The standard is analyzed under the appropriate method and compared to the existing inventory of standards. If the response of any target species is not within acceptance limits for a second source standard, the standard is considered to have failed. The vendor is then contacted to discuss the discrepancy and arrangements made to replace the standard.

**Exhibit 12.1 Critical Suppliers**

<b>VENDOR</b>	<b>JUSTIFICATION</b>
Scott Specialty Gases	Sole supplier/Reliable service over several years
Airgas	Reliable service over several years
Spectra Gases Inc.	Reliable service
SKC West Inc.	Sole supplier
Chromatography Research Supplies, Inc.	Sole supplier
Scientific Instrument Services	Reliable service over several years
Hamilton	Reliable service over several years
Quantum Analytics, Inc.	Reliable service over several years
R & D Glassware	Reliable service over several years
Aldrich Chemical Company	NIST Certified
Supelco	Reliable service over several years
Valco	Sole Supplier
VWR	Reliable service over several years
Control Company	Reliable service
Agilent Technologies	Reliable service
Millipore/Mykrolis	Reliable service over several years
Leco Corporation	Sole Supplier
Oakland Valve & Fitting CO.	Sole Supplier/Reliable Service

### **13.0 PROJECT MANAGEMENT SYSTEM**

Any quality system, no matter how elaborate, and no matter how well documented, will fail unless the customer expectation is effectively communicated. The ATL Project Management System describes the critical pathways necessary to adequately ensure that the customer expectation is reviewed, committed to by top management, documented and communicated to the laboratory prior to sample delivery. System elements include:

- Review of project specific documents
- Negotiations and variance requests
- Documentation of project requirements
- Documenting client discussions
- Project briefings
- Scheduling sampling media
- Tracking sample analysis and reporting
- Project follow-up

The Project Management System is defined in the ATL SOP #1. Following are brief descriptions of the elements comprising project management systems.

#### **13.1 REVIEW OF PROJECT SPECIFIC DOCUMENTS**

Clients document project requirements in requests for proposals, work plans, SOWs, or QAPPs. No matter how the details are documented, the project requirements must be reviewed to ensure that the laboratory has sufficient staff, equipment, and capacity to meet project needs. Any document received from a client containing either contractual language, description of work, QA/QC criteria, and/or deliverable requirements different from our Standard Terms and Conditions will be processed as a proposal. When a proposal is received, an electronic

Proposal File is started and routed through the Proposal Tracking System using the c.Support software. The Account Managers are provided with technical support by the designated Project Chemist who reviews all relevant sections of the proposals. The Department Managers are consulted for further information when needed.

Items to be reviewed include:

- Requested methods
- Adequate and documented training of appropriate personnel including Demonstration of Capability
- Adequate instrumentation
- Target compound lists and reporting limits
- Quantity of samples
- Requested media and degree of preparation
- Media preservation requirements
- Holding time requirements
- Project specific QC
- Deliverable requirements
- TAT requirements
- Insurance requirements
- Special billing and payment terms
- Data storage requirements
- Client financial status

The following flow chart (Exhibit 13.1) depicts how the proposal is distributed through the review process and project requirements are received and logged into a database. Each proposal is given a unique identifier by the Account Manager. The Account Manager determines project feasibility, consulting with the Technical Director when necessary. The Account Manager reviews the document to determine whether or not the proposal contains methods, target compounds, or production demands requiring additional review and delegates sections to the appropriate department. If necessary, the

Project Chemist and IT Manager review the document for special project requirements in the areas of:

- Quality assurance criteria
- Reporting criteria
- Unusual compounds
- Electronic deliverable criteria
- Unusual Media Requirements
- Unusual volume of samples

If the Project Chemist has any questions, which may affect production, then the Account Manager is notified and the proposal is sent to the Technical Director to determine the implications to the laboratory production throughput. If the proposal does not require method development, and has unusual deliverables then it is sent to the IT Manager or designee to verify if ATL is able to meet client deliverable demands. The reviewer may suggest pricing adjustments according to the client specific format difficulties. If the proposal does not require review by the IT Manager, it is returned to the Account Manager. A variance table to be included with the proposal is created by the Project Chemist. The variance table may undergo revision as per the result of discussion with the client.

The Contract specifications are reviewed by the Contract Administrator to evaluate the terms and conditions of the proposal, including the following areas:

- Retention;
- Penalties;
- Sales and Use Tax Requirements;
- Insurance Requirements;
- Data Storage Requirements; and
- Payments Terms.

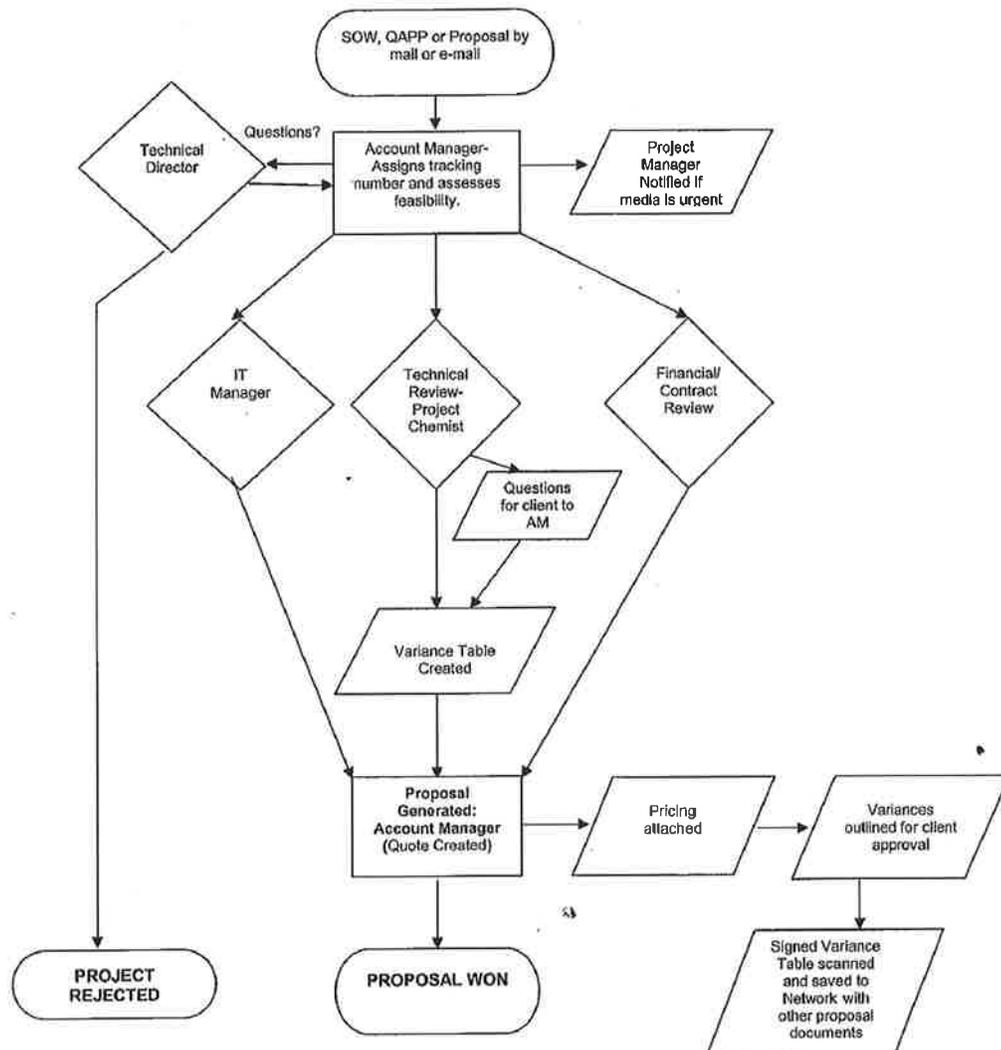
The Contract Administrator then reviews the proposal to determine the amount of credit to be established for the client. The amount will

be based upon credit status (Dunn & Bradstreet) report, the amount of the contract and past payment history with ATL.

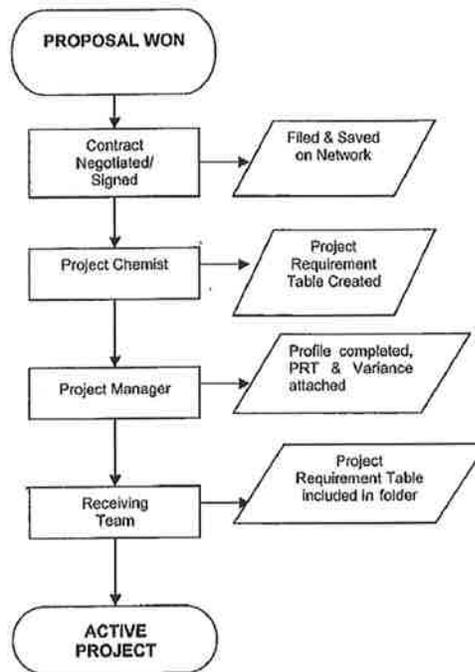
Following review and comments, the proposal is returned to the Account Manager for final review and pricing. The proposal is then entered into the ATLAS Quotes module for reference. If the bidding process is successful, the Project Manager will set up an active project, utilizing the proposal documents and quote as references. Before media is shipped, a signed copy of the variance table must be scanned to the network. The Account Manager and Project Chemist are brought back at this time if negotiations of the variance table are necessary. The Project Manager will complete the project profile, while the Project Chemist creates a project requirement table using the approved variance table which is stored on the network drive. An example of project profile appears in Section 3, Exhibit 3.3.

Before the final report is released a contract must be signed and filed. Once a contract is received, it is documented in the contract log. The contract is then reviewed to compare the pricing and language to the proposal. If there are exceptions made to the contract, these are noted and negotiated with the client. Once pricing and terms have been agreed upon, the contract is then executed and copies sent back to the client. If the client needs to amend the contract after the project has started the contract review process is performed for the amended contract. When changes to the contract affects any current procedures (i.e. invoicing, analysis, reporting) the appropriate personnel are notified via e-mail and associated documents are updated. In addition, ATL will notify the client of any suspensions, revocations, or voluntary withdrawals of accreditation resulting from the amended contract.

Exhibit 13.1 Proposal Review Flow Chart



**Exhibit 13.1 Proposal Review Flow Chart (Continued)**



### **13.2 NEGOTIATIONS AND VARIANCE REQUESTS**

When the Project Chemist notes differences between the project request and laboratory standard protocol, the laboratory may request a variance from the requirements. Ideally, variance requests occur during the proposal stage, but sufficient details regarding project requirements are sometimes not known until sampling is about to begin. With the assistance of the QA team the Project Chemist (or designee) notes all discrepancies in a variance table that is submitted to the Account Manager. The assigned Account Manager communicates the discrepancies to the client by submitting the variance table along with the proposal. Variance requests are most often handled directly between these parties. On occasion, a conference call may be held with an agency representative or additional project and laboratory staff present. It is the responsibility of the Account Manager to coordinate the meeting.

Once an agreement has been reached and the variance table is approved by the client, the Project Chemist will finalize the understandings concerning the discrepancies in a project requirement table, which is used for sample login and analysis. The variance table and project requirement table are given unique identifiers that reference the unique identifier assigned to the proposal. The tables are stored on a network-shared drive in read-only format until sample log in occurs.

### **13.3 DOCUMENTATION OF PROJECT REQUIREMENTS**

The Project Manager becomes the primary client contact following the project award. The Project Manager will verify at that time that the client has been provided most recent version of the project plans. Any outstanding issues are discussed by phone and documented in the Client Contacts database. At this time the project profile is updated to include:

- Shipping information
- Report To and Bill To information
- Pricing
- Reference method
- Requested media and degree of preparation
- Target compound list
- Special QA requirements
- Calibration criteria (if different from SOP)
- Deliverable requirements
- Account Information, including payment terms
- Variance table by reference
- Project Requirement table by reference
- Any special instructions
- Subcontracting (when relevant)

The project profiles are secured with read only privileges for all staff except the Project Managers, Account Managers, Department Managers, the Project Chemist and members of the QA team.

### **13.4 DOCUMENTING CLIENT DISCUSSIONS**

Once a project has been awarded, the majorities of contacts occur via phone or e-mail and are documented in the Project Management software. The software can track contacts by client name, project name, or date. Project Management team members may sort the database for summaries of calls made on the basis of specific clients or sub-contracting entities.

### **13.5 DOCUMENTING CLIENT CONCERNS**

Client concerns are documented in the Project Management software. Project Managers respond quickly to address client concerns within 24 hours. Resolution of these concerns is also documented in the Project Management software. Concerns of a more serious nature are documented utilizing a Corrective Action Report procedure. These are maintained and

by the QA Department. The Project Manager will follow up with the client once the corrective action has been implemented and the report is closed.

### **13.6 SCHEDULING SAMPLING MEDIA**

The ATL Project Managers work closely with clients to ensure that media is delivered according to project schedule. Shipments for on-going projects are scheduled well in advance. The Project Managers are responsible for processing shipping requests through the Client Contacts database. This is the same database used to log phone contacts. Each shipping request is given a unique identifier. Both laboratory staff and shipping staff monitor the database throughout the day for posted shipping requests.

Some media types (e.g., DNPH solutions, PUF/XAD cartridges, etc.) take time to prepare. Careful planning and scheduling on the part of both the Project Managers and the clients is necessary. Clients are encouraged to provide as much lead-time as possible.

### **13.7 TRACKING SAMPLE ANALYSIS AND REPORTING**

The sample tracking database is reviewed daily and provides the Project Manager with up-to-date information on sample status. Daily contact with the Team Leader or Department Managers is necessary when the rate of the production is less than desirable in any area. It is the responsibility of the Project Manager to inform the client within 24 hours in the event that delays in analysis and reporting are anticipated and deviations from project requirement and/or contract agreements have occurred.

### **13.8 PROJECT FOLLOW-UP**

All contact with the client following reporting is handled by the Project Managers. This includes requests for re-issues, perceived problems with data, or further clarification.

All discussions are documented in the Client Contacts database within 24 hours. When a client desires a modification to a completed report, the Project Manager reviews the request. Commonly the Department Manager responsible for the work or the QA Manager may be consulted. If the request is considered to be legitimate, the Project Manager initiates a request for report re-issue (per ATL SOP #68) in the Sample Tracking database. The database tracks the re-issue status to make sure that the report is fixed and sent to the client. The reason for the re-issue is documented on the report cover along with report re-issue date. It is the responsibility of the Project Manager to ensure that the correct reason and supporting documentation is provided in the report folder.

**This page left blank intentionally**

## 14.0 DATA INTEGRITY PROCEDURES

### 14.1 TRAINING

Data integrity is the cornerstone of Air Toxics Limited. Our Mission Statement and Value Statement define the goals and values which produce data of known and defensible quality. Everything from data reporting programs, employee retention programs to customer service programs evolve around maintaining our core values above all else.

#### ATL Mission Statement

- To deliver high quality cost effective environmental analytical services
- That are profitable, on time and meet/exceed the expectations of our customers

How we go about accomplishing this mission is governed by our standards of conduct; the ability to discern right from wrong and proper from improper and the commitment to always do what is right, good and proper.

***“We seek success in what we do, but not at the expense of integrity. Integrity is essential to establishing and maintaining the trust that allows us to work as a team and to foster confidence in our customers and co-workers. Without integrity there can be no trust.”***

Without integrity, our customers would not trust us to get the job done and would not hire us. Employees would not trust management to set realistic goals and provide adequate resources. Without integrity, management would not trust employees with the ultimate form of empowerment.... direct responsibility for customer satisfaction.

All new employees are provided with introductory training which consists of:

#### Ethics and Integrity Training I

The ATL Value Statement  
The ATL Mission Statement  
Definition of Standards of Conduct and Ethics  
ATL Strategic Goals  
Importance of Trust  
Definition of Integrity  
Four basic enemies of integrity with workplace examples  
Criminal ramifications for misconduct  
Benefits of Integrity Training  
Role of the Employee (reporting systems)  
Role of Management (promote core values)  
Where to find ATL Codes of Conduct  
Employee Handbook (employment standards)  
Quality Manual (quality standards)  
SOPs (technical standards)  
Chemical Hygiene Plan (safety standards)

A yearly refresher course is provided which reviews the basic information of Ethics and Integrity Training I but then goes on to define the critical steps in making ethical decisions:

#### Ethics and Integrity Training II

1. Review relevant values and standards, devise a plan.
2. Question the plan, your motives and any consequences.
3. Resolve to address any ethical dilemmas by making tough choices.

Case studies are used in Ethics and Integrity Training II to encourage work group participation in actual decision making scenarios. Following each training session, the employee is provided with a certificate for training and asked to sign the certificate indicating they have been informed of their obligations in the ATL integrity program and understand that legal ramifications may be imposed upon them for failure to comply.

#### **14.2 PERIODIC MONITORING**

There are three parts to the ongoing periodic monitoring for inappropriate data manipulations following initial training. First, the IT Department has removed access to adjustment of data acquisition and reporting computer clocks. Second, each new employee undergoes a training period in which all of their data is reviewed by a Scientist or Team Leader until such time as basic knowledge and comprehension of method as well as data integrity procedures is demonstrated. A development plan is provided to each new employee by their Department Manager or Team Leader, which outlines goals, timelines and demonstration of understanding. The measurement of success may include proficiency with a task as well as a verbal or written test demonstrating concept comprehension.

When the Department Manager and/or Team Leader determine the new employee is ready to produce data of defensible quality, he/she is asked to provide several representative data packages to the Quality Assurance Manager for thorough review. If the QA Manager agrees that defensible data was produced, the employee is released from the 100% review program.

The third and final part of the periodic monitoring program involves the use of proprietary in-house data validation software to review every data point generated and to alert the reviewer when manual integrations occur. The software is also programmed to report when an analyte in the initial calibration and QC samples does not meet established acceptance criteria. (Validation software currently reviews all method TO-14A/TO-15, TO-17, ASTM D-5504, TO-3, TO-12 and SW8260B results. Further software development is currently ongoing to bring more methods on line).

#### **14.3 MECHANISMS FOR REPORTING INFRACTIONS**

During Ethics and Integrity Training I, the new employee is informed how to go about communicating any real or perceived infractions of the data integrity system. Open dialogue is encouraged between the employee and any member of management or Senior Scientists they feel most comfortable with. It is management's responsibility to relay the information to the Technical Director(s) and follow-up with an inquiry or corrective action. The employee who desires to remain anonymous is encouraged to report to the team's Senior Scientist as ombudsman. The Senior Scientist will meet separately with management and the employee involved in order to ensure anonymity. An immediate inquiry by one of the Technical Directors will follow each and every reported incident. Documentation of the inquiry and subsequent disciplinary actions will be maintained by both the Technical Director and the Human Resource Department for at least 5 years.

## Appendix A

### DEFINITIONS AND TERMS

**Accuracy:** The degree of agreement between an observed value and an accepted reference value.

**Analyte:** The substance or thing for which a sample is analyzed to determine its presence or quantity.

**Approved:** The determination by any state for federal accrediting agency that a certified laboratory may analyze for an analyte under the specified method.

**Assessment:** The process of inspecting, testing and documenting findings for purposes of certification or to determine compliance.

**Batch:** A group of analytical samples ( $\leq 20$ ) of the same matrix processed together including extraction, concentration and analysis using the same process, staff and reagents.

**Bag:** Means an inert air-sampling container consisting of inert polymeric material.

**Canister:** A stainless steel spherical air-sampling device consisting of summa polished or glass lined internal walls and a leak tight on/off valve.

**Contamination:** The effect caused by the introduction of the target analyte from an outside source into the test system.

**Continuing Calibration Verification (CCV):** A CCV is analyzed to verify instrument linearity with respect to the Initial Calibration. The CCV concentration may be identical to any given point contained within the initial calibration. A CCV is analyzed at the beginning of every analytical sequence (all methods) and then once every ten or twenty samples depending on the method (GC and LC, GC/MS excluded). GC and LC methods also include a CCV in every analytical sequence as an End Check.

**Control Charts:** These are statistical tools for monitoring the performance of a particular task on a continuing basis. The control chart is prepared for each test parameter after 20 determinations have been performed. The mean is plotted with the warning limits being  $\pm 2s$  and the control limits being  $\pm 3s$ . ( $s$  – standard deviation)

**Corrective Action:** An action taken to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

**Data Reduction:** A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data are of acceptable quality.

**Duplicate Samples:** Samples collected for checking the preciseness of the sampling process. These samples are collected at the same time and from the same source as the study samples.

**Equipment Blank:** A sample that is known not to contain the target analyte that is used to check the cleanliness of sampling devices, collected in a sampling container from a clean sample collection device and returned to the laboratory as a sample.

## Appendix A

### DEFINITIONS AND TERMS

**Field Blank:** A sample that is known not to contain the target analyte and is used to check for analytical artifacts or contamination introduced by sampling and analytical procedures, carried to the sampling site, exposed to sampling conditions and returned to the laboratory and treated as an environmental sample.

**Field Duplicate:** Samples collected at the same time from the same source, but submitted and analyzed as separate samples.

**Holding Time:** The maximum time that a sample may be held prior to preparation or analysis.

**Impinger:** The glass vessel used to contain collection solution through which a stream of air is bubbled for sampling purposes.

**Initial Calibration:** Demonstration of a linear response to different concentrations of calibration standards within a defined range.

**Initial Demonstration of Analytical Capability:** The procedure described in the method 40 CFR Appendix A, used to determine a laboratory's accuracy and precision in applying an analytical method.

**Instrument Blank:** A sample that is known not to contain the target analyte, processed through the instrumental steps of the measurement process used to determine the absence of instrument contamination prior to analysis of field samples.

**Instrument Detection Limit (IDL):** It is the concentration of the analyte that produces a signal greater than five times the signal-to-ratio of the instrument.

**Interference:** The effect on the final result caused by the sample matrix.

**Internal Standards (ISs):** These are the measured amounts of certain compounds added after preparation or extraction of a sample.

**Key Personnel:** The laboratory director, technical director, quality assurance manager, and team leader, all of whom meet the requirements of the NELAP rule.

**Laboratory Control Sample:** An independent second source reference standard which goes through the same pretreatment and preparation procedures as the samples. It validates the accuracy of the initial calibration.

**Laboratory Duplicates:** Aliquots of the same sample that are prepared and analyzed at the same time.

**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. The LOD may be determined by a Method Detection Limit study.

**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. Generally, the LOQ is equal to the concentration of analyte(s) in the lowest point of a calibration (see Reporting Limit).

## Appendix A

### DEFINITIONS AND TERMS

**Matrix:** The component or substrate (e.g., surface water, drinking water, air, liquid waste) which contains the analyte of interest.

**Matrix Spike:** A sample prepared to determine the effect of the matrix on a method's recovery efficiency by adding a known amount of the target analyte to a specified amount of matrix sample for which an independent estimate of the target analyte concentration is available.

**Matrix Spike Duplicate (MSD):** Duplicates of the matrix spike sample.

**Measurement Uncertainty:** Measurement uncertainty is the estimation of potential errors in a measurement process and is expressed as  $\pm 2X (s)$  of the historical mean of LCS recoveries.

**Method Detection Limit:** The minimum concentration of a substance that can be measured and reported with 99% confidence that the analyte concentration is greater than zero as determined from analysis of a sample containing the analyte in a given matrix (40 CFR Part 136, Appendix B, July 1995).

**Practical Quantitation Limit (PQL):** A synonym for the standard of lowest concentration contained in the Initial Calibration. It is the smallest concentration of the analyte that can be reported with a specific degree of confidence.

**Precision:** The degree to which a set of observations or measurements of the same property, obtained under similar conditions, conform to themselves. Precision is usually expressed as standard deviation, variance or arrange, in either absolute or relative terms.

**Preservation:** The temperature control or the addition of a substance to maintain the chemical or biological integrity of the target analyte.

**Proficiency Testing (PT) Assessment:** The event including receiving, analyzing, and reporting of results from a set of samples that a proficiency testing provider sends to a laboratory for the laboratory to demonstrate compliance with the proficiency testing requirements.

**Proficiency Test (PT) Sample:** 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.

**Quality Assurance:** An integrated system of activities involving planning, quality control, reporting, and quality assessment and improvement to ensure that the product meets defined standards of quality with a stated level of confidence.

**Quality Assurance Project Plan (QAPP):** An orderly assemblage of detailed procedures designed to produce data of sufficient quality to meet the data quality objectives for a specific data collection activity.

**Reporting Limit:** The smallest concentration of an analyte that can be measured with a stated probability of significance. All Initial Calibrations contain a standard at the Reporting Limit. The Reporting Limit is never less than the PQL.

## Appendix A

### DEFINITIONS AND TERMS

**Reporting Limit Verification:** A re-quantification of the lowest concentration data point of an initial calibration to test the percent recovery of each component. Analyte recovery should be between 50-150% to verify detection limit accuracy.

**Selectivity:** The capability of a method or instrument to respond to the target analyte in the presence of other substances or things.

**Sensitivity:** The capability of a method or instrument to discriminate between measurement responses representing different levels of a target analyte.

**Standard Operating Procedures (SOP):** A written document which details the steps of an operation, analysis or action whose techniques and procedures are thoroughly prescribed, and is accepted as the procedure for performing certain routine or repetitive tasks.

**Surrogate:** A substance which is unlikely to be found in the environment and which has properties that mimic the target analyte and that is added to a sample to check for analytical efficiency.

**Target Analyte:** The analyte that a test is designed to detect or quantify.

**Technical Employee:** A designated individual who performs the analytical method and associated techniques.

**Trip Blank:** A sample known not to contain the target analyte that is carried to the sampling site and transported to the laboratory for analysis without having been exposed to the sampling procedures.

## Appendix B

### STANDARD OPERATING PROCEDURES

#1	Customer Support System
#2	Analysis of Volatile Organic Compounds in VOST Cartridges and Condensates by Modified EPA SW-846 Method 5041A/8260B
#3	Analysis of Semi-Volatile Organic Compounds by Modified EPA SW-846 Method 8270C
#4	Preparation and Conditioning of VOST and CMS Tubes
#5	Analysis of Volatile Organic Compounds in Ambient Air Using Modified EPA Method TO-17 with Modified EPA SW-846
#6	Analysis of Volatile Organic Compounds in SUMMA™ Polished Canisters by Modified EPA Methods TO-14A/TO-15
#7	Preparation of Silonite™, Silcosteel™, and Summa™ Canisters for Sampling
#8	Analysis of Oxygen, Nitrogen, Methane, Ethane, Ethene, Carbon Monoxide, Carbon Dioxide, Hydrogen and NMOC by Modified ASTM Method D-1946
#10	Analysis of Semi-Volatile Organic Compounds Collected on PUF Cartridges by GC/MS Full Scan Modified EPA Method TO-13A
#11	Analysis of Aldehydes and Ketones by Modified EPA Methods TO-5 (Impingers), TO-11A (Sep-Pak Cartridges), Modified SW-846 Method 0011/8315A (Impingers) and Modified CARB 430 (Impingers)
#12	Extraction of Aldehydes and Ketones by Modified EPA Method TO-5 and Modified CARB 430
#13	Analysis of Sulfur Compounds by ASTM Method D-5504
#14	Preparation of PUF and PUF/XAD-2 Media for Ambient Air Sampling and XAD-2 Media for MM5 Sampling
#15	Extraction of Ambient Air & MM-5 Samples for Semivolatile Analysis and Pesticides/PCB Analysis by Modified EPA SW-846 Method 3542
#17	Safe Lifting Procedures
#19	Refrigerator/Freezer Temperature Monitoring and Documentation
#20	ATL GC Applications for Analysis of Organic Compounds in Air
#24	Storage and Disposal of Hazardous Wastes
#25	Extraction of Aldehydes by Modified Methods 0011 and 8315A
#26	Analysis of Pesticides and PCBs Collected on PUF Cartridges using Modified EPA Methods TO-4A/TO-10A
#27	Internal Audit Procedures
#30	Laboratory Security

## Appendix B

### STANDARD OPERATING PROCEDURES

#33	Standard Preparation, Validation, and Documentation
#34	Certification of Test Equipment
#36	Analysis of Non-Methane Organic Compounds (NMOC) Using Modified EPA Method TO-12
#38	Analysis of Volatile Organic Compounds in Summa™ Polished Canisters by GC/MS Selective Ion Monitoring Modified EPA Methods TO-14A/TO-15
#39	Procedures to Perform an MDL Study
#43	Analysis of Benzene, Toluene, Ethylbenzene, Xylene and Total Petroleum Hydrocarbons in Ambient Air Using Modified EPA Method TO-3
#44	Certification of SUMMA™ Polished or Glass-Lined Canisters used in the Analysis of Volatile Organic Compounds
#45	Preparation and Review of Laboratory Narratives
#46	Writing and Updating Standard Operating Procedures
#47	Implementation and Testing of Atlas Modules
#48	Preparation and Review of Control Charts
#50	Receipt and Tracking of Samples
#52	Manual Peak Integration for GC/MS Analyses
#53	Tune Check Spectrum Generation
#54	Analysis of Natural Gases by Modified ASTM Method D-1945
#55	Electronic Archival of Analytical Instrument Data
#57	Manual Peak Integration - Gas Chromatography
#59	Screening Samples Using GC/FID
#60	Canister Pressurization
#61	Corrective Action Procedure
#62	Preparation of 2,4-Dinitrophenylhydrazine (DNPH) Reagent for Use in Modified EPA Methods 0011, TO-5, TO-11A, and CARB 430 Sampling Media
#63	Sample Custody Cage Logbook Documentation
#65	Extraction of Aldehydes by Modified EPA Method TO-11A
#66	Determination of Suspended Particulate Matter in the Atmosphere as Total Suspended Particulates from Quartz Fiber Filters by 40 CFR Part 50 Appendix B; Determination of Particulate Matter as PM10 from Quartz Fiber Filters by 40 CFR Part 50 Appendix J

## Appendix B

### STANDARD OPERATING PROCEDURES

#68	Procedure to Reissue Finalized Data Reports
#69	Transfer of Sample Collected in a Tedlar Bag to a Summa™ Canister
#70	Preparation and Procedures for Flow Controller Assemblies
#71	Siloxanes in Air by GC/MS Direct Inject Analysis
#74	Analysis Of Polycyclic Aromatic Hydrocarbons by GC/MS Selective Ion Monitoring (SIM) Modified EPA Method TO-13A
#78	Generation of Air Toxics Ltd. Data Deliverables, Electronic Conversion, and Archival
#79	Analysis of Volatile Organic Compounds in SMVOC Cartridges (Tenax®-GC and Anasorb® 747) and Condensates by Modified EPA SW-846 Method 5041A/8260B /0031
#83	Analysis Of Volatile Organic Compounds In Summa™ Polished Canisters by GC/MS Low Level Modified EPA Methods TO-14A/TO-15
#87	Demonstration of Capability Procedure
#89	Media Management
#90	Subcontract Analysis
#91	Analysis Of Volatile Organic Compounds In Summa™ Polished Canisters by GC/MS Modified EPA Methods TO-14A/TO-15 (5 & 20 ppbv)
#92	Analysis Of Volatile Organic Compounds In Soil Gas Collected in Passivated Canisters by EPA SW-846 Modified Method 8260B
#93	Method Editor Procedure
#94	Valid Values Editor and Compound Editor Procedure
#95	Client Canister Tracking System
#96	ATLAS ( Air Toxics Ltd. Automated System)
#97	Client Inquiry Procedure
#98	Instrument Set Up Procedure For New Gas Chromatography/Mass Spectrometer(GC/MS) Or Relocating An Existing GC/MS
#100	Analysis Of Volatile Organic Compounds Collected On Charcoal-Based Passive Samplers

## REFERENCES

- Annual Book of ASTM Standards, American Society for Testing & Materials, 1991.
- EPA Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air (First and Second Editions)
- EPA Compendium of Methods for the Determination of Air Pollutants in Indoor Air, Winberry *et al.*, Atmospheric Research and Exposure Assessment Laboratory, September 1989.
- Environmental Sampling and Analyses Lab Manual, Maria Csuros, Lewis Publishers, 1997.
- Test Methods for Evaluating Solid Waste, SW-846, Third Edition, Final Update III, Revision 1, December, 1996.
- Statement of Work for the Organic Superfund Contract Laboratory Program, OLM04.2 May, 1999.
- CLP Draft - Analytical Method for the Determination of VOCs in Air Collected in Summa Canisters and Analyzed by GC/MS, December 1991.
- Guidelines Establishing Test Procedures for the Analysis of Pollutants under the Clean Water Act 40 CFR (136) 1984.
- NIOSH Manual of Analytical Methods, Center for Disease Control, Fourth Edition, August, 1994.
- OSHA Analytical Methods Manual, U.S. Department of Labor, January, 1990.
- California Air Resources Board Stationary Source Test Manual, Monitoring and Laboratory Division, September, 1990.

**7.0 TO-14A/TO-15 – VOLATILE ORGANIC COMPOUNDS**

This method involves full scan GC/MS analysis of whole air samples collected in evacuated stainless steel canisters. Samples are analyzed for volatile organic compounds using EPA Method TO-14A/TO-15 protocols. An aliquot of the sample is withdrawn from the canister through a mass flow controller and is either concentrated using a cryogenic trap and/or concentrated using a hydrophobic multisorbent bed. The hydrophobic multisorbent bed functions as a drying system which removes water from the sample stream prior to analysis by full scan GC/MS. During analysis, the sample may be focused onto a cryogenic cooled column and/or a cryogenic cooled sleeve for analysis by full scan GC/MS.

Certain compounds are not included in ATL’s standard target analyte list. These compounds are communicated at the time of client proposal request. Unless otherwise directed, ATL reports these non-standard compounds with partial validation. Validation includes a 3-point calibration with the lowest concentration defining the reporting limit, no second source verification is analyzed, and no method detection limit study is performed unless previous arrangements have been made. In addition, stability of the non-standard compound during sample storage is not validated. Full validation may be available upon request.

Air Toxics Ltd. performs a modified version of this method. The method modifications, standard target analyte list, Limit of Quantitation, QC criteria, and QC summary can be found in the following tables.

**Table 7-1. Summary of Method Modifications**

<b>Requirement</b>	<b>TO-14A</b>	<b>TO-15</b>	<b>Air Toxics Ltd. Modifications</b>
Sample Drying System	Nafion Drier.	Multisorbent.	Multisorbent.
Blank acceptance criteria	< 0.2 ppbv.	< RL.	< RL.
Blanks and standards ( <b>applies to Low Level analysis only</b> )	Zero Air.	Zero air.	Nitrogen.
BFB absolute abundance criteria	Within 10% of that from the previous day.	Not mandated.	CCV internal standard area counts are compared to ICAL, corrective action for > 40 %D.
Method Detection Limit	Not Specified.	Follow 40CFR Pt.136 App. B.	The MDL met all relevant requirements in Method TO-15 (statistical MDL less than the LOQ). The concentration of the spiked replicate may have exceeded 10X the calculated MDL in some cases.

Requirement	TO-14A	TO-15	Air Toxics Ltd. Modifications
Initial Calibration	≤ 30 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % RSD.	≤ 30 % RSD with 2 compounds allowed out to ≤ 40 % for <b>QUAD and 5&amp;20</b> analysis and 4 compounds allowed out to ≤ 40 % for <b>Low Level</b> analysis.
Daily CCV	≤ 30% D.	≤ 30% D.	For <b>QUAD and 5&amp;20</b> analysis: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.  For <b>Low Level</b> analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.
Sample collection media.	Summa canister.	Summa canister.	Methods TO-14A/TO-15 are validated for samples collected in specially treated canisters. As such, the use of Tedlar bags for sample collection is outside the scope of these methods and not recommended for ambient or indoor air samples. Associated results are considered qualified.

**Table 7-2. Method TO-14A/TO-15 Analyte List**

Analyte	RL (ppbv) TO-15/ LL/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits (Max. RPD)
1,1,2,2-Tetrachloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1,2-Trichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,1-Dichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2,4-Trichlorobenzene	2.0/0.5/20	30%	70 - 130	≤ 25
1,2,4-Trimethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dibromoethane (EDB)	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,2-Dichloropropane	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,3,5-Trimethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,3-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
1,4-Dichlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Benzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Bromomethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
Carbon Tetrachloride	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chlorobenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloroethane	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloroform	0.5/0.1/5.0	30%	70 - 130	≤ 25
Chloromethane	2.0/0.1/20	30%	70 - 130	≤ 25
Chlorotoluene (Benzyl Chloride)	0.5/0.1/5.0	30%	70 - 130	≤ 25
cis-1,2-Dichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
cis-1,3-Dichloropropene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Dichloromethane	0.5/0.2/5.0	30%	70 - 130	≤ 25
Ethylbenzene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 11 (Trichlorofluoromethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 113 (Trichlorotrifluoroethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 114	0.5/0.1/5.0	30%	70 - 130	≤ 25
Freon 12 (Dichlorodifluoromethane)	0.5/0.1/5.0	30%	70 - 130	≤ 25
Hexachlorobutadiene	2.0/0.5/20	30%	70 - 130	≤ 25
m,p-Xylene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Methyl Chloroform	0.5/0.1/5.0	30%	70 - 130	≤ 25
o-Xylene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Styrene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Tetrachloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Toluene	0.5/0.1/5.0	30%	70 - 130	≤ 25
trans-1,3-Dichloropropene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Trichloroethene	0.5/0.1/5.0	30%	70 - 130	≤ 25
Vinyl Chloride	0.5/0.1/5.0	30%	70 - 130	≤ 25

**Table 7-3. Method TO-14A/TO-15 Analyte List**

Analyte	RL (ppbv) TO-15/ LL/5&20	%RSD	Acceptance Criteria	
			LCS (%R)	Precision Limits
1,3-Butadiene	0.5/0.1/5.0	30%	60 – 140	≤ 25
1,4-Dioxane	2.0/0.1/20	30%	60 – 140	≤ 25
2-Butanone (Methyl Ethyl Ketone)	0.5/0.1/5.0	30%	60 – 140	≤ 25
2-Hexanone	2.0/0.5/20	30%	60 – 140	≤ 25
4-Ethyltoluene	0.5/0.1/5.0	30%	60 – 140	≤ 25
4-Methyl-2-Pentanone (MIBK)	0.5/0.1/20	30%	60 – 140	≤ 25
Acetone	2.0/0.5/20	30%	60 – 140	≤ 25
Bromodichloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Bromoform	0.5/0.1/5.0	30%	60 – 140	≤ 25
Carbon Disulfide	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cyclohexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Dibromochloromethane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Ethanol	2.0/0.5/20	30%	60 – 140	≤ 25
Heptane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Hexane	0.5/0.1/5.0	30%	60 – 140	≤ 25
Isopropanol	2.0/0.5/20	30%	60 – 140	≤ 25
Methyl t-Butyl Ether (MTBE)	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylene	2.0/0.5/20	30%	60 – 140	≤ 25
Tetrahydrofuran	0.5/0.5/5.0	30%	60 – 140	≤ 25
trans-1,2-Dichloroethene	0.5/0.1/5.0	30%	60 – 140	≤ 25
2,2,4-Trimethylpentane	0.5/0.5/5.0	30%	60 – 140	≤ 25
Cumene	0.5/0.1/5.0	30%	60 – 140	≤ 25
Propylbenzene	0.5/0.1/5.0	30%	60 – 140	≤ 25
3-Chloroprene	2.0/0.5/20	30%	60 – 140	≤ 25
Naphthalene	2.0/0.5/20	30%	60 – 140	≤ 25
TPH (Gasoline) or NMOC (Hexane/Heptane)	10/2.0/50	One Point Calibration	NA	≤ 25

**Table 7-4. Internal Standards**

Analyte	Accuracy (% R)
Bromochloromethane	60 - 140
1,4-Difluorobenzene	60 - 140
Chlorobenzene-d <sub>5</sub>	60 - 140

**Table 7-5. Surrogates**

Analyte	Accuracy (% R)
1,2-Dichloroethane-d <sub>4</sub>	70 – 130
Toluene-d <sub>8</sub>	70 – 130
4-Bromofluorobenzene	70 – 130

**Table 7-6. Summary of Calibration and QC Procedures for Methods TO-14A/TO-15**

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Tuning Criteria	Every 24 hours, or every 12 hours if project requires.	SW – 846 tune criteria.	Correct problem then repeat tune.
5-Point Calibration	Prior to sample analysis.	% RSD $\leq$ 30 with two compounds allowed out to $\leq$ 40% RSD for QUAD and 5&20 (4 allowed out for LL).	Correct problem then repeat Initial Calibration Curve.
LCS	After each initial calibration curve, and daily, prior to sample analysis.	Recoveries for 90% of "Standard" compounds must be 70-130%; for 80% of "Non-standard" compounds, recoveries must be 60-140%. No recovery may be <50%.  * If specified by the client in-house generated control limits may be used.	Check the system and reanalyze the standard. Re-prepare the standard if necessary. Re-calibrate the instrument if the criteria cannot be met.
Continuing Calibration Verification (CCV)	At the start of each day and, if required by a specific project, every 12 hours.	For QUAD and 5&20: 70-130%. Compounds exceeding this criterion and associated data will be flagged and narrated with the exception of high bias associated with non-detects. If more than two compounds from the standard list recover outside of 70-130%, corrective action will be taken. Unless prior client approval; under no circumstances will samples be analyzed if any compound exceeds 60-140%.  For <b>Low Level</b> analysis the above applies except corrective action will be taken if more than four compounds from the standard list recover outside of 70-130%.	Perform maintenance and repeat test. If the system still fails the CCV, perform a new 5 point calibration curve.
Laboratory	After the CCV/LCS.	Results less than the	Inspect the system and

QC Check	Minimum Frequency	Acceptance Criteria	Corrective Action
Blank		laboratory reporting limit.	Re-analyze the blank.
Internal Standard (IS)	As each standard, blank, and sample is being loaded.	Retention time (RT) for blanks and samples must be within $\pm 0.33$ min of the RT in the CCV and within $\pm 40\%$ of the area counts of the daily CCV internal standards.	<b>For blanks:</b> inspect the system and reanalyze the blank. <b>For samples:</b> re-analyze the sample. If the ISs are within limits in the re-analysis, report the second analysis. If ISs are out-of-limits a second time, dilute the sample until ISs are within acceptance limits and narrate.
Surrogates	As each standard, blank, and sample is being loaded.	70 - 130%.  * If specified by the client in-house generated control limits may be used.	<b>For blanks:</b> inspect the system and reanalyze the blank. <b>For samples:</b> re-analyze the sample unless obvious matrix interference is documented. If the %R is within limits in the re-analysis, report the second analysis. If %R is out-of-limits a second time, then narrate results.
Laboratory Duplicates	10% of the samples.	RPD $\leq 25\%$ for detections >5 X's the RL.	Re-analyze the sample a third time. If the limit is exceeded again, investigate the cause and bring the system back to working order. If no problem is found on the system, narrate results.

## Reporting Limits for Site Specific VOCs

Compound	Reporting Limit (ppbv)	Reporting Limit (ug/m3)	MDL (ppbv)	MDL (ug/m3)
Vinyl Chloride	0.5	1.3	0.06	0.15
1,1-Dichloroethene	0.5	2.0	0.07	0.27
trans-1,2-Dichloroethene	0.5	2.0	0.18	0.72
1,1-Dichloroethane	0.5	2.0	0.07	0.29
cis-1,2-Dichloroethene	0.5	2.0	0.11	0.45
1,1,1-Trichloroethane	0.5	2.7	0.04	0.23
Benzene	0.5	1.6	0.04	0.12
Trichloroethene	0.5	2.7	0.09	0.46
Toluene	0.5	1.9	0.05	0.19
Tetrachloroethene	0.5	3.4	0.09	0.58
Ethyl Benzene	0.5	2.2	0.06	0.27
m,p-Xylene	0.5	2.2	0.06	0.25
o-Xylene	0.5	2.2	0.05	0.22

ARCADIS

**Attachment 3**

**TestAmerica Analytical Testing Corp.**

Quality Assurance Manual, 2008

NCMS019 Method 8260B, 8260A, 2009

Corporate Quality Management Plan, 2009

Canprt70, 2007

# Cover Page:

## Quality Assurance Manual

TestAmerica North Canton  
4101 Shuffle Street NW  
North Canton, OH 44720  
Phone: 330-497-9396  
Fax: 330-497-0772  
[www.testamericainc.com](http://www.testamericainc.com)

**Copyright Information:**

This documentation has been prepared by TestAmerica Analytical Testing Corp. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

**THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:  
©COPYRIGHT 2007 TESTAMERICA ANALYTICAL TESTING CORP. ALL RIGHTS RESERVED**

Facility Distribution No. \_\_\_\_\_

Distributed To: \_\_\_\_\_

**Title Page:**

**Quality Assurance Manual  
Approval Signatures**



Laboratory Director – Opal Davis-Johnson

12/10/07

Date



Quality Manager – Dorothy Leeson

12/10/07

Date



Technical Director – Dr. Mark Bruce

12/10/07

Date

**SECTION 2**  
**TABLE OF CONTENTS**

<b>Section No.</b>	<b>Title</b>	<b>Page No.</b>	<b>Effective Date</b>
-	COVER PAGE	COVER	01/01/2008
1.0	<b>TITLE PAGE</b>	1-1	01/01/2008
2.0	TABLE OF CONTENTS	2-1	01/01/2008
3.0	INTRODUCTION	3-1	01/01/2008
3.1	Introduction And Compliance References	3-1	01/01/2008
3.2	Terms And Definitions	3-1	01/01/2008
3.3	Scope / Fields Of Testing	3-1	01/01/2008
3.4	Management Of The Manual	3-2	01/01/2008
4.0	ORGANIZATION AND MANAGEMENT ( <i>NELAC 5.4.1</i> )	4-1	01/01/2008
4.1	Overview	4-1	01/01/2008
4.2	Roles And Responsibilities	4-2	01/01/2008
5.0	QUALITY SYSTEM ( <i>NELAC 5.4.2</i> )	5-1	01/01/2008
5.1	Quality Policy Statement	5-1	01/01/2008
5.2	Ethics And Data Integrity	5-1	01/01/2008
5.3	Quality System Supporting Documentation	5-2	01/01/2008
5.4	Qa/Qc Objectives For The Measurement Of Data	5-3	01/01/2008
5.5	Criteria For Quality Indicators	5-5	01/01/2008
5.6	Statistical Quality Control	5-5	01/01/2008
5.7	Quality System Metrics	5-6	01/01/2008
6.0	DOCUMENT CONTROL ( <i>NELAC 5.4.3</i> )	6-1	01/01/2008
6.1	Overview	6-1	01/01/2008
6.2	Document Approval And Issue	6-1	01/01/2008
6.3	Procedures For Document Control Policy	6-2	01/01/2008
6.4	Obsolete Documents	6-2	01/01/2008
7.0	REVIEW OF WORK REQUEST	7-1	01/01/2008
7.1	Overview	7-1	01/01/2008
7.2	Review Sequence And Key Personnel	7-2	01/01/2008
7.3	Documentation	7-3	01/01/2008
8.0	SUBCONTRACTING OF TESTS ( <i>NELAC 5.4.5</i> )	8-1	01/01/2008
8.1	Overview	8-1	01/01/2008
8.2	Qualifying And Monitoring Subcontractors	8-2	01/01/2008
8.3	Oversight And Reporting	8-5	01/01/2008
8.4	Contingency Planning	8-6	01/01/2008
9.0	PURCHASING SERVICES AND SUPPLIES ( <i>NELAC 5.4.6</i> )	9-1	01/01/2008
9.1	Overview	9-1	01/01/2008

Section No.	Title	Page No.	Effective Date
9.2	Glassware	9-1	01/01/2008
9.3	Reagents, Standards & Supplies	9-1	01/01/2008
9.4	Purchase Of Equipment/Instruments/Software	9-3	01/01/2008
9.5	Services	9-4	01/01/2008
9.6	Suppliers	9-4	01/01/2008
10.0	SERVICE TO THE CLIENT <i>(NELAC 5.4.7)</i>	10-1	01/01/2008
10.1	Overview	10-1	01/01/2008
10.2	Special Services	10-1	01/01/2008
10.3	Client Communication	10-1	01/01/2008
10.4	Reporting	10-1	01/01/2008
10.5	Client Surveys	10-2	01/01/2008
11.0	COMPLAINTS <i>(NELAC 5.4.8)</i>	11-1	01/01/2008
11.1	Overview	11-1	01/01/2008
11.2	External Complaints	11-1	01/01/2008
11.3	Internal Complaints	11-2	01/01/2008
11.4	Management Review	11-2	01/01/2008
12.0	CONTROL OF NON-CONFORMING WORK <i>(NELAC 5.4.9)</i>	12-1	01/01/2008
12.1	Overview	12-1	01/01/2008
12.2	Responsibilities And Authorities	12-1	01/01/2008
12.3	Evaluation Of Significance And Actions Taken	12-2	01/01/2008
12.4	Prevention Of Nonconforming Work	12-2	01/01/2008
12.5	Method Suspension/Restriction (Stop Work Procedures)	12-2	01/01/2008
13.0	CORRECTIVE ACTION <i>(NELAC 5.4.10)</i>	13-1	01/01/2008
13.1	Overview	13-1	01/01/2008
13.2	Definitions	13-1	01/01/2008
13.3	General	13-1	01/01/2008
13.4	Closed Loop Corrective Action Process	13-2	01/01/2008
13.5	Technical Corrective Actions	13-3	01/01/2008
13.6	Basic Corrections	13-3	01/01/2008
14.0	PREVENTIVE ACTION <i>(NELAC 5.4.11)</i>	14-1	01/01/2008
14.1	Overview	14-1	01/01/2008
14.2	Management Of Change	14-2	01/01/2008
15.0	CONTROL OF RECORDS <i>(NELAC 5.4.12)</i>	15-1	01/01/2008
15.1	Overview	15-1	01/01/2008
15.2	Technical And Analytical Records	15-5	01/01/2008
15.3	Laboratory Support Activities	15-6	01/01/2008
15.4	Administrative Records	15-6	01/01/2008
15.5	Records Management, Storage And Disposal	15-6	01/01/2008
16.0	AUDITS <i>(NELAC 5.4.13)</i>	16-1	01/01/2008
16.1	Overview	16-1	01/01/2008
16.2	Technical And Analytical Records	16-1	01/01/2008
16.3	External Audits	16-3	01/01/2008
16.4	Audit Findings	16-5	01/01/2008
17.0	MANAGEMENT REVIEWS <i>(NELAC 5.4.14)</i>	17-1	01/01/2008

Section No.	Title	Page No.	Effective Date
17.1	Quality Assurance Report	17-1	01/01/2008
17.2	Annual Management Review	17-2	01/01/2008
17.3	Potential Integrity Related Managerial Reviews	17-3	01/01/2008
18.0	PERSONNEL <i>(NELAC 5.5.2)</i>	18-1	01/01/2008
18.1	Overview	18-1	01/01/2008
18.2	Education And Experience Requirements For Technical Personnel	18-1	01/01/2008
18.3	Training	18-3	01/01/2008
18.4	Data Integrity And Ethics Training Program	18-4	01/01/2008
19.0	ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS <i>(NELAC 5.5.3)</i>	19-1	01/01/2008
19.1	Overview	19-1	01/01/2008
19.2	Environment	19-1	01/01/2008
19.3	Work Areas	19-2	01/01/2008
19.4	Floor Plan	19-2	01/01/2008
19.5	Building Security	19-2	01/01/2008
20.0	TEST METHODS AND METHOD VALIDATION <i>(NELAC 5.5.4)</i>	20-1	01/01/2008
20.1	Overview	20-1	01/01/2008
20.2	STANDARD OPERATING PROCEDURES (Sops)	20-1	01/01/2008
20.3	Laboratory Methods Manual	20-1	01/01/2008
20.4	Selection Of Methods	20-2	01/01/2008
20.5	Laboratory Developed Methods And Non-Standard Methods	20-16	01/01/2008
20.6	Validation Of Methods	20-16	01/01/2008
20.7	Method Detection Limits (Mdl)/ Limits Of Detection (Lod)	20-18	01/01/2008
20.8	Instrument Detection Limits (Idl)	20-19	01/01/2008
20.9	Verification Of Detection And Reporting Limits	20-20	01/01/2008
20.10	Retention Time Windows	20-20	01/01/2008
20.11	Evaluation Of Selectivity	20-21	01/01/2008
20.12	Estimation Of Uncertainty Of Measurement	20-21	01/01/2008
20.13	Control Of Data	20-22	01/01/2008
21.0	EQUIPMENT (AND CALIBRATIONS) <i>(NELAC 5.5.5)</i>	21-1	01/01/2008
21.1	Overview	21-1	01/01/2008
21.2	Preventive Maintenance	21-1	01/01/2008
21.3	Support Equipment	21-3	01/01/2008
21.4	Instrument Calibrations	21-5	01/01/2008
21.5	Policy On Tentatively Identified Compounds (Tics) – Gc/Ms Analysis	21-13	01/01/2008
21.6	Policy On Gc/Ms Tuning	21-13	01/01/2008
22.0	MEASUREMENT TRACEABILITY <i>(NELAC 5.5.6)</i>	22-1	01/01/2008
22.1	Overview	22-1	01/01/2008
22.2	Nist-Traceable Weights And Thermometers	22-2	01/01/2008
22.3	Reference Standards / Materials	22-2	01/01/2008

Section No.	Title	Page No.	Effective Date
22.4	Documentation And Labeling Of Standards, Reagents, And Reference Materials	22-3	01/01/2008
23.0	SAMPLING <i>(NELAC 5.5.7)</i>	23-1	01/01/2008
23.1	Overview	23-1	01/01/2008
23.2	Sampling Containers	23-1	01/01/2008
23.3	Field Quality Control (Qc)	23-1	01/01/2008
23.4	Definition Of Holding Time	23-2	01/01/2008
23.5	Sampling Containers, Preservation Requirements, Holding Times	23-3	01/01/2008
23.6	Sample Aliquots / Subsampling	23-3	01/01/2008
24.0	HANDLING OF SAMPLES <i>(NELAC 5.5.8)</i>	24-1	01/01/2008
24.1	Chain Of Custody (Coc)	24-1	01/01/2008
24.2	Sample Receipt	24-2	01/01/2008
24.3	Sample Acceptance Policy	24-4	01/01/2008
24.4	Sample Storage	24-4	01/01/2008
24.5	Hazardous Samples And Foreign Soils	24-5	01/01/2008
24.6	Sample Shipping	24-5	01/01/2008
24.7	Sample Disposal	24-5	01/01/2008
25.0	ASSURING THE QUALITY OF TEST RESULTS <i>(NELAC 5.5.9)</i>	25-1	01/01/2008
25.1	Overview	25-1	01/01/2008
25.2	Controls	25-1	01/01/2008
25.3	Negative Controls	25-1	01/01/2008
25.4	Positive Controls	25-2	01/01/2008
25.5	Sample Matrix Controls	25-4	01/01/2008
25.6	Acceptance Criteria (Control Limits)	25-6	01/01/2008
25.7	METHOD DETECTION LIMITS (Mdl)	25-8	01/01/2008
25.8	Additional Procedures To Assure Quality Control	25-8	01/01/2008
26.0	REPORTING RESULTS <i>(NELAC 5.5.10)</i>	26-1	01/01/2008
26.1	Overview	26-1	01/01/2008
26.2	Test Reports	26-1	01/01/2008
26.3	Reporting Level Or Report Type	26-3	01/01/2008
26.4	Supplemental Information For Test	26-4	01/01/2008
26.5	Environmental Testing Obtained From Subcontractors	26-4	01/01/2008
26.6	Client Confidentiality	26-4	01/01/2008
26.7	Format Of Reports	26-5	01/01/2008
26.8	Amendments To Test Reports	26-5	01/01/2008
26.9	Policies On Client Requests For Amendments	26-5	01/01/2008

## LIST OF TABLES

<b>Table No.</b>	<b>Title</b>	<b>Page</b>	<b>Effective Date</b>
9-1	<u>Storage of Reagents and Chemicals</u>	9-6	01/01/2008
13-1	<u>Example - General Corrective Action Procedures</u>	13-5	01/01/2008
15-1	<u>Records and Retention Schedule</u>	15-1	01/01/2008
15-2	<u>Special Record Retention Requirements</u>	15-3	01/01/2008
16-1	<u>Audit Types and Frequency</u>	16-1	01/01/2008
20-1	<u>Wet Chemistry Methods</u>	20-3	01/01/2008
20-2	<u>Methods for Mercury by Cold Vapor Atomic Absorption</u>	20-7	01/01/2008
20-3	<u>Methods for Mercury by Cold Vapor Atomic Fluorescence</u>	20-7	01/01/2008
20-4	<u>Methods for Metals by ICP and ICPMS</u>	20-7	01/01/2008
20-5	<u>Metals Sample Preparation Methods</u>	20-11	01/01/2008
20-6	<u>Organic Sample Preparation Methods</u>	20-12	01/01/2008
20-7	<u>Organic Method of Analysis</u>	20-14	01/01/2008
21-1	<u>Example - Laboratory Equipment &amp; Instrumentation</u>	21-15	01/01/2008
21-2	<u>Example – Laboratory Balance Inventory</u>	21-19	01/01/2008
21-3	<u>Example – Laboratory Refrigerators, Freezers, Ovens, Thermometers, Steambaths, Hotblocks, and Hotplates</u>	21-20	01/01/2008
21-4	<u>Example: Schedule of Routine Maintenance</u>	21-22	01/01/2008
21-5	<u>Example: Periodic Equipment Calibrations</u>	21-32	01/01/2008
21-6	<u>Example: Preventive Maintenance Procedures</u>	21-33	01/01/2008

<b>Table No.</b>	<b>Title</b>	<b>Page</b>	<b>Effective Date</b>
23-1	<u>Inorganic Sample Containers, Preservatives, and Holding Times</u>	23-5	01/01/2008
23-2	<u>Organic Sample Containers, Preservatives, and Holding Times</u>	23-15	01/01/2008
23-3	<u>Sample Containers, Preservatives, and Holding Times for TCLP and SPLP</u>	23-23	01/01/2008

## LIST OF FIGURES

Figure No.	Title	Page	Effective Date
3-1	<u>Example - Format for a QA/QC Policy Memorandum</u>	3-4	01/01/2008
4-1	<u>Corporate Organizational Chart</u>	4-9	01/01/2008
8-1	<u>Example - Client-Approved Subcontractor Form</u>	8-7	01/01/2008
8-2	<u>Example - Subcontracting Laboratory Approval Form (Initial / Renewal)</u>	8-8	01/01/2008
9-2	<u>Example - JD Edwards Vendor Add Request Form</u>	9-7	01/01/2008
16-1	<u>Example - Internal Audit Workbook</u>	16-7	01/01/2008
16-2	<u>Example – Internal Audit System Checklist</u>	16-8	01/01/2008
17-1	<u>Example - QA Monthly Report to Management</u>	17-4	01/01/2008
17-2	<u>Example – Laboratory Metrics Categories</u>	17-6	01/01/2008
20-1	<u>Example – Demonstration of Capability Documentation</u>	20-31	01/01/2008
20-2	<u>Example – New Method / Additional Analyte Checklist</u>	20-32	01/01/2008
20-3	<u>Work Flow</u>	20-33	01/01/2008
24-1	<u>Example: Chain of Custody (COC)</u>	24-7	01/01/2008
24-2	<u>Example: Custody Seals</u>	24-8	01/01/2008
24-3	<u>Example: Internal Chain of Custody (COC)</u>	24-9	01/01/2008
24-4	<u>Example: Cooler Receipt Form</u>	24-11	01/01/2008

## LIST OF APPENDICES

Appendix No.	Title	Page	Effective Date
1	<u>TestAmerica Ethics Policy No. CA-L-P-001</u>	Appendix 1-1	01/01/2008
2	<u>Example - Laboratory Organization Chart</u>	Appendix 2-1	01/01/2008
3	<u>Laboratory Floor Plan</u>	Appendix 3-1	01/01/2008
4	<u>Laboratory Method Listing</u>	Appendix 4-1	01/01/2008
5	<u>Laboratory Reporting Limits</u>	Appendix 5-1	01/01/2008
6	<u>Glossary/Acronyms</u>	Appendix 6-1	01/01/2008
7	<u>Laboratory Certifications, Accreditations, Validations</u>	Appendix 7-1	01/01/2008
8	<u>Data Qualifiers</u>	Appendix 8-1	01/01/2008

## SOPs AND POLICIES REFERRED TO IN THE QA MANUAL

SOP/Policy Reference	Title
CA-C-S-001	Work Sharing Process
CA-L-S-001	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-P-001	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CA-L-S-002	Subcontracting Procedures
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-003	Management of Change Procedure
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-005	Calibration Curves (General)
CA-T-P-001	Qualified Products List
CORP-MS-0001NC	GC/MS Analysis Based on Method 8270C,
CORP-MS-0002NC	Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).
CORP-QA-0010	Nonconformance and Corrective Action System
CORP-QA-0013	Employee Orientation and Training
CW-F-P-002	Authorization Matrix
CW-F-S-004	Controlled Purchases Policy
CW-L-P-001	Record Retention
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
NC-QA-0012	Shipping Department
NC-QA-015	Equipment Monitoring and Thermometer Calibration
NC-QA-0017	Standards and Reagents
NC-QA-0018	Statistical Evaluation of Data and Development of Control Charts
NC-QA-0019	Records Information Management
NC-QA-0020	Laboratory Holding Blanks
NC-QA-0021	Evaluation of Method Detection Limits for Chemical Tests

NC-QA-0027	Preparation and Management of Standard Operating Procedures
NC-SC-0005	Sample Receiving and Sample Control
NC-SC-0006	Sample Procurement Protocol
QA-003	TestAmerica North Canton Quality Control Program
S-C-002	Complaint Handling and Service Recovery
S-Q-001	Document Control
S-Q-004	Acceptable Manual Integration Practices

## SECTION 3

### INTRODUCTION (NELAC 5.1 - 5.3)

#### 3.1 INTRODUCTION AND COMPLIANCE REFERENCES

The TestAmerica North Canton Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica data quality goals. Each TestAmerica 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 the various accreditation and certification programs listed in Appendix 7. 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 600/4-79-019, *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.
- U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- Toxic Substances Control Act (TSCA).

#### 3.2 TERMS AND DEFINITIONS

A Quality Assurance Program is a company-wide system designed to ensure that data produced by TestAmerica North Canton 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 6 for the Glossary/Acronyms.

#### 3.3 SCOPE / FIELDS OF TESTING

TestAmerica analyzes thousands of environmental and industrial samples every month. Sample matrices vary among effluent water, groundwater, hazardous waste, sludge, wipes, 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 water, industrial waste, and soil 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. 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 TestAmerica North Canton 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, the Quality Assurance (QA) Manager, and the Technical Director. 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.

- Specific requirements delineated in project plans may supersede general quality requirements described in this manual. Ohio VAP requirements are listed throughout the document.

### **3.4 MANAGEMENT OF THE MANUAL**

#### **3.4.1 Review Process**

The manual is reviewed annually by the QA Manager and laboratory personnel to assure that it reflects current practices and meets the requirements of TestAmerica North Canton clients and regulators. 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. The updates will be reviewed by the QA Manager, Laboratory Director/Manager, Technical Director(s), relevant operational staff and Corporate Quality Assurance (if a change is made to the Corporate template) and then formally incorporated into the document in periodic updates. The QAM is based on a Corporate QAM Template that is prepared and approved by the Chief Operating Officers (COOs) and Corporate Quality Assurance. This template is reviewed annually by the COOs, Corporate Quality, and each laboratory. Necessary changes are coordinated by the Vice President of Quality and Environmental Health & Safety (EHS) and distributed to each laboratory for inclusion in the laboratory specific QA Manuals.

Policies in the QAM that require immediate attention may be addressed through the use of Corporate QA/QC Policy Memoranda. QA/QC Policy Memoranda are published from time to time to facilitate immediate changes to QA/QC Policy. QA/QC Policy Memoranda supersede the QAM and all other SOPs (refer to Section 5.3). All policy memoranda are dated, archived and distributed by their placement into the front of the QAM between the signature page and Section 2. At a minimum, each policy memorandum is approved by the same authorized signatories as shown on the cover page of the QA Manual. In addition, Corporate QA/QC Policy

Memoranda are signed by the COOs and VP of Quality and EHS. The QA/QC Policy Memoranda are incorporated into the QAM during the periodic updates. Policy memorandum may also include an expiration date if appropriate. An example format can be found in Figure 3-1. A similar procedure is followed for local laboratory changes.

### **3.4.2      Control**

This manual is considered confidential within TestAmerica and may not be altered in any manner by other than a duly appointed representative from TestAmerica. If the document has been provided to external users or regulators, it is for the exclusive purpose of reviewing TestAmerica North Canton quality systems and shall not be used in any other way without the written permission of an appointed representative of TestAmerica. The procedure for control of distribution is incorporated by reference to SOP S-Q-001, Document Control.

The order of precedence in the event of a conflict between policies is outlined in Section 5.3 of this Quality Assurance Manual.

**Figure 3-1.**

**Example - Format for a QA/QC Policy Memorandum**

<b>TestAmerica North Canton</b>	
<b>SOP/LQM CHANGE FORM</b>	
SOP/LQM NUMBER:	
TITLE:	
SECTION(S) AFFECTED BY CHANGE:	
REASON FOR ADDITION OR CHANGE:	
CHANGE EFFECTIVE FROM: (DATE):	
CHANGE OR ADDITION:	
SUBMITTED BY/DATE:	
*APPROVED BY:	
Technical Reviewer Signature	Date
Environmental Health & Safety Signature	Date
QA Signature	Date
Technical Director (if applicable)	Date
Laboratory Director Signature	Date

## SECTION 4

### ORGANIZATION AND MANAGEMENT (NELAC 5.4.1)

#### 4.1 OVERVIEW

TestAmerica North Canton is part of a national network of laboratories known as TestAmerica. This Quality Assurance Manual (QAM) is applicable to the TestAmerica North Canton laboratory only.

**TestAmerica North Canton Laboratory**  
**4101 Shuffle Drive NW**  
**North Canton, OH 44720**  
**EPA I.D. Code No. 9503**

The Corporate organization chart can be found in Figure 4-1 and the laboratory organization chart can be found in Appendix 2. The locations of other TestAmerica labs are as follows:

Aerotech Environmental Laboratories (AEL)  
TestAmerica Anchorage  
TestAmerica Austin  
TestAmerica Buffalo  
TestAmerica Buffalo Grove  
TestAmerica Burlington  
TestAmerica Cedar Falls  
TestAmerica Chicago  
TestAmerica Colorado Springs  
TestAmerica Connecticut  
TestAmerica Corpus Christi  
TestAmerica Dayton  
TestAmerica Denver  
TestAmerica Edison  
TestAmerica Honolulu  
TestAmerica Houston  
TestAmerica Irvine  
TestAmerica King of Prussia  
TestAmerica Knoxville  
TestAmerica Los Angeles  
TestAmerica Mobile  
TestAmerica Morgan Hill  
TestAmerica Nashville  
TestAmerica New Orleans  
TestAmerica Ontario  
TestAmerica Orlando  
TestAmerica Pensacola  
TestAmerica Phoenix  
TestAmerica Pittsburgh

TestAmerica Portland  
TestAmerica Richland  
TestAmerica San Francisco  
TestAmerica Savannah  
TestAmerica Seattle  
TestAmerica Spokane  
TestAmerica St. Louis  
TestAmerica Tacoma  
TestAmerica Tallahassee  
TestAmerica Tampa  
TestAmerica Valparaiso  
TestAmerica Watertown  
TestAmerica West Sacramento  
TestAmerica Westfield

## **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 define each role in its relationship to the Quality Assurance Program. More extensive job descriptions are maintained by laboratory management.

### **4.2.1 Quality Assurance Program**

The responsibility for quality lies with every employee of TestAmerica North Canton. All employees have access to the QAM and are responsible for knowing the content of this manual and 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.

### **4.2.2 Chairman/Chief Executive Officer (CEO)**

The Chairman/CEO is the Chairman of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the President/CEO of the Analytical Division, the Chairman/CEO establishes the overall quality standard and data integrity program for the company, providing the necessary leadership and resources to assure that the standard and integrity program are met.

### **4.2.3 President/Chief Executive Officer (CEO)**

The President/CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. Together with the Chairman/CEO, the President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met.

#### **4.2.4 Chief Operating Officer (COO) – East and West**

The COOs serve as the ranking executives for all respective analytical laboratory operational functions and report to the President/CEO of the Analytical Division. They are responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. They ensure the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COOs approve all operating budgets and capital expenditures. The COOs sign-off on the final QAM template that contains company policies for implementing the Quality Program.

#### **4.2.5 General Manager (GM)**

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.

#### **4.2.6 Vice President of Quality and Environmental Health and Safety (VP-QA/EHS)**

The Vice President of QA/EHS reports directly to the Chairman/CEO. With the aid of the Analytical Division and Non-Analytical Division Senior Management Teams, Laboratory Director/Managers, Quality Directors, EHS Directors, QA Managers and EHS Coordinators, the VP-QA/EHS has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs, national projects and expansions or changes in services.
- Coordination/preparation of the Corporate QAM Template that is used by each laboratory to prepare its own laboratory-specific QAM.
- Maintenance of Corporate Policies, Quality Memorandums and SOPs. Maintenance of data investigation records that are reported to Corporate Management.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.
- With the assistance of the Corporate Senior Management Teams and the EHS Directors, development and implementation of the TestAmerica Environmental, Health and Safety Program.

#### **4.2.7 Quality Directors (Corporate)**

The Quality Directors report to the VP-QA/EHS. Together with the VP-QA/EHS, the Quality Directors have the responsibility for the establishment, general overview and maintenance of

the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a final review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification activities.

#### **4.2.8 Ethics and Compliance Officers (ECOs)**

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP-QA/EHS and VP-Client and Technical Services. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECO is responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEOs, COOs, Laboratory Director/Manager or other appropriate individuals within the laboratory. The ECO will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

#### **4.2.9 Vice President of Client and Technical Services**

The Vice President (VP) of Client and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Client and Technical Services provides support and direction to the Managers of these areas, and supports the COOs in decisions regarding long term planning, resource allocation and capital expenditures.

#### **4.2.10 Director of Technical Services**

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

#### **4.2.11 Chief Information Officer (CIO)**

The CIO is responsible for establishing, implementing and communicating TestAmerica Information Technology (IT) Policies, SOPs, and Manuals. Other responsibilities include coordinating new technologies; development of electronic communication tools such as TestAmerica intranet and internet sites; ensuring data security and documentation of software; ensuring compliance with the NELAC standard; and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

#### **4.2.12 Environmental Health and Safety Directors (EHSDs) (Corporate)**

The EHSDs report directly to the VP-QA/EHS. The EHSDs are responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the corporate Environmental, Health and Safety Manual Template that is used by each laboratory to prepare its own laboratory-specific Safety Manual/ CHP.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

#### **4.2.13 Laboratory Director**

- Reports directly to the Regional General Manager
- Responsible for implementation and adherence by lab staff to the TestAmerica North Canton QAM and all policies and procedures within the laboratory
- Has signature authority for QAM, policies, SOPs, and contracts (as detailed in TestAmerica policy)
- Assesses the effectiveness of the QAM within the operation
- Maintains adequate trained staff documented on organization charts
- Responsible for implementing internal/external audit findings corrective actions

#### **4.2.14 Quality Assurance Manager**

- Reports directly to the Laboratory Director and, for all QA matters, to the Corporate QA Director to maintain independence of QA oversight
- Responsible for the implementing and communicating the QAM
- Maintains, approves, and implements the QAM

- Has joint signature authority, with the Laboratory Director and Technical Director for approval of quality documents, e.g., QAM, policies, and SOPs
- Directs controlled distribution of laboratory quality documents
- Provides Quality System training to all new personnel
- Reviews and approves documentation of analyst training records
- Serves as a focal point for QA and QC issues, reviews corrective actions and recommends resolution for recurring nonconformances within the laboratory
- Assists in maintaining regulatory analytical compliance, including maintaining certifications, and in this regard has signature authority for laboratory quality documents
- Monitors data quality measures via statistical methods to verify that the laboratory routinely meets stated quality goals
- Performs systems, data, contract compliance, and surveillance audits.
- Hosts external audits conducted by outside agencies
- Responsible for approving quality control reference data changes in the LIMS
- Oversees the selection, review, and approval of analytical subcontractors
- Prepares monthly QA Reports to management describing significant quality events
- Has the final authority to accept or reject data and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data
- Responsible for implementing internal/external audit findings corrective actions

#### **4.2.15 Operations Manager/Laboratory Supervisor**

- Reports directly to the Laboratory Director
- Supervises daily activities of the Operational Groups
- Schedules analytical operations
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Supervises maintenance of instruments and scheduling of repairs
- Works with the Project Managers and Group Leaders to assure the requirements of projects are met in a timely manner
- Supervises daily activities of the Sample Control Group.

#### **4.2.16 Laboratory Technical Director**

- Reports directly to Laboratory Director
- Responsible for the technical operation of the laboratory
- Has joint signature authority for QAM, policies, SOPs, and training records
- Performs technical training in area(s) of expertise
- Interfaces with management on technical needs and solving day-to-day technical issues
- Investigates technical issues related to projects as directed by QA
- Evaluates new methods, technical proposals, and statements of work
- Certifies technical laboratory personnel based on education and background to ensure that staff have demonstrated capability in the activities for which they are responsible by reviewing and signing analyst demonstrations.
- Performs other tasks as required by NELAC.

The Technical Director meets the requirements specified in Section 4.1.1.1 of NELAC standards.

#### **4.2.17 Project Management Group Leader**

- Reports directly to the Manager of Client Services
- Supervises daily activities of the Project Management and Administrative Groups
- Works with the Operations Manager and/or Group/Team Leaders to ensure the requirements of projects are met in a timely manner

#### **4.2.18 Manager of Client Services**

- Reports directly to Laboratory Director
- Supervises Field Analytical Services Group
- Supervises Service Centers
- Supervises daily activities of the customer
- Supervises Service Managers

#### **4.2.19 Customer Service Managers (CSMs)**

- Reports directly to the Manager of Client Services
- Defines customer requirements through project definition
- Assesses and assures customer satisfaction
- Provides feedback to management on changing customer needs
- Brings together resources necessary to ensure customer satisfaction.

#### **4.2.20 Project Manager**

- Reports directly to the Project Manager Group Leader
- Monitors analytical and QA project requirements for a specified project
- Acts as a liaison between the client and the laboratory staff
- Communicates project-specific requirements to all parties involved
- Assists the laboratory staff with interpretation of work plans, contracts, and QAPP requirements
- Oversees project data packages for completeness and compliance to client needs
- Has signature authority for final reports
- Keeps the laboratory and client informed of project status
- Together with the QA Manager, approves customer requested variances to methods and to standard laboratory protocols
- Monitors, reviews, and evaluates the progress and performance of projects
- Reports client inquiries involving data quality issues or data acceptability to the facility QA Manager and to the operations staff
- Conducts project reviews to assess the laboratory's performance in meeting customer requirements
- Prepares reissue requests for project data
- Responsible for meeting quality requirements.

#### **4.2.21 Group Leader**

- Reports directly to the Operations Manager
- Supervises daily activities of analyses within the group
- Supervises QC activities performed as a part of routine analytical operations
- Implements data review procedures
- Supervises the preparation and maintenance of laboratory records
- Evaluates instrument performance and supervises the calibration, preventive maintenance, and scheduling of repairs
- Oversees or performs review and approval of all analytical data
- Reports nonconformances to the appropriate managers
- Responsible for generation of SOPs for their section
- Responsible for meeting quality requirements.

#### **4.2.22 Analyst**

- Performs analytical methods and data recording in accordance with documented procedures
- Performs and documents calibration and preventive maintenance
- Performs data processing and data review procedures
- Reports nonconformances to the Supervisor/Manager and QA Manager
- Ensures sample and data integrity by adhering to internal chain-of-custody procedures
- Responsible for meeting quality requirements defined in this LQM and other supporting QA procedures.

#### **4.2.23 Sample Custodian**

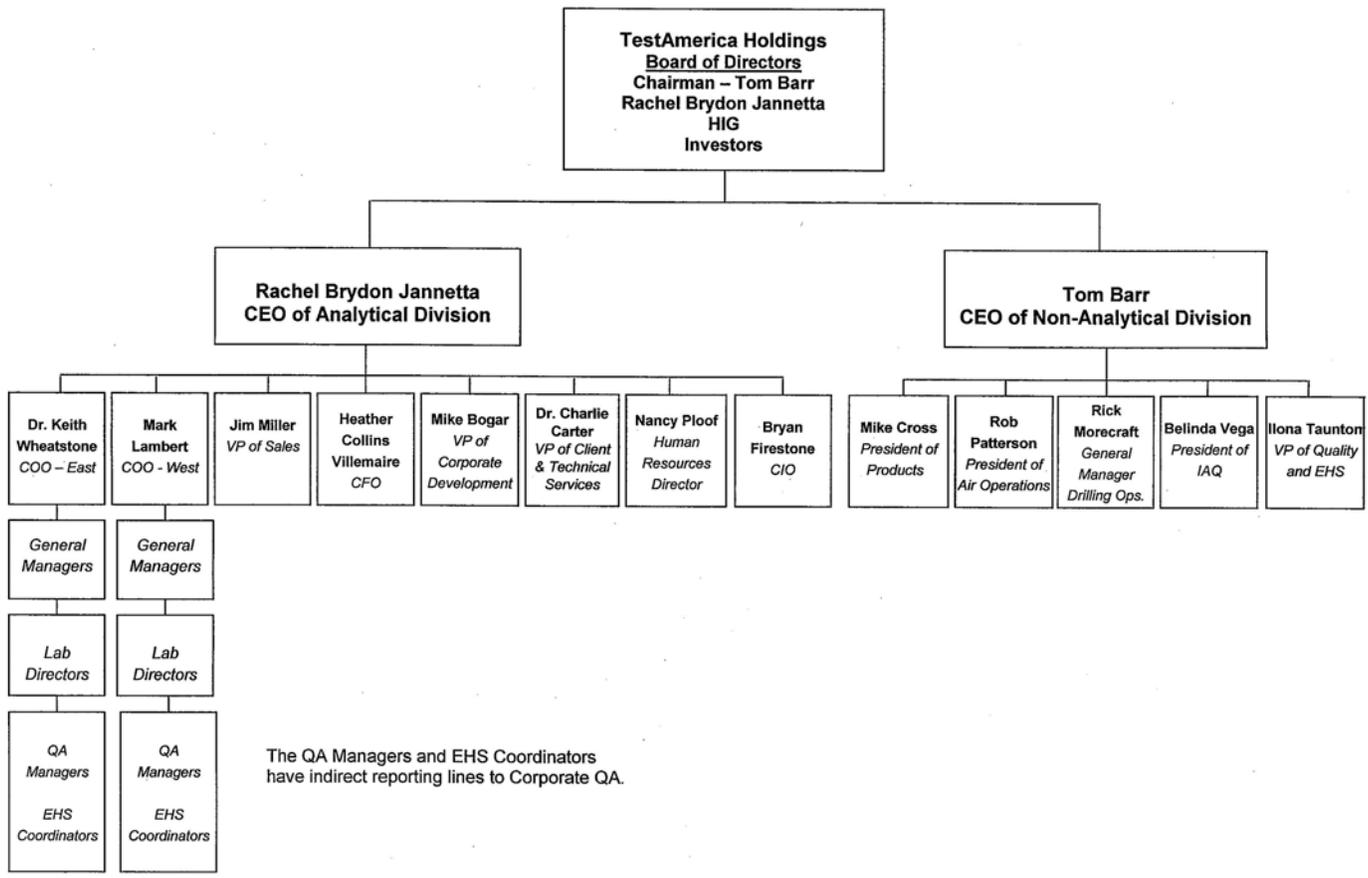
- Ensures implementation of proper sample receipt procedures, including maintenance of chain-of-custody
- Reports nonconformances associated with condition-upon-receipt of samples
- Logs samples into the LIMS
- Ensures that all samples are stored in the proper environment
- Assists Environmental Health and Safety staff with sample disposal
- Responsible for meeting quality requirements.

#### **4.2.24 Report Production Staff**

- Accurately generates and compiles analytical reports and associated deliverables for delivery to the client
- Responsible for meeting quality requirements
- Produce as needed reports that meet the NELAC requirements.

Figure 4-1.

Corporate Organization Chart



## SECTION 5

### QUALITY SYSTEM (NELAC 5.4.2)

#### 5.1 QUALITY POLICY STATEMENT

The management of TestAmerica and TestAmerica North Canton are committed to providing data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols described in this manual.

In all aspects of the laboratory and business operations, management is dedicated in maintaining the highest ethical standards. An Ethics Policy sign-off can be viewed in Appendix 1. Training on ethical and legal responsibilities is provided annually and each employee signs off annually on the policy as a condition of employment.

It is TestAmerica's Policy to continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. The company recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.

TestAmerica North Canton strives to provide clients with the highest level of professionalism and the best service practices in the industry.

Every staff member at TestAmerica North Canton 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 seven elements of the TestAmerica Ethics and Data Integrity Program include:

- An Ethics Policy (Policy CA-L-P-001) and Employee Ethics Statements (Appendix 1)
- An Ethics and Compliance Officer (ECO)
- 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 (SOP CA-L-S-001)
- Procedures and guidance for recalling data if necessary (SOP CA-L-S-001)
- An effective external and internal monitoring system that includes procedures for internal audits (Section 16)

As an American Council of Independent Laboratories (ACIL) member, all TestAmerica laboratories adhere to the following ACIL Code of Ethics:

- Produce results, which are accurate and include QA/QC information that meets client pre-defined 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 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 SUPPORTING DOCUMENTATION**

The Laboratory Quality System is communicated through a variety of documents prepared by the laboratory and company management:

- Quality Assurance Manual (QAM) Template
- Quality Assurance Manual – Each laboratory has a lab specific quality assurance manual.
- Corporate SOPs and Policies - 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.
- Work Instructions - 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
- Corporate TestAmerica QA/QC Policy Memorandums (refer to Section 3.4).
- Laboratory QA/QC Policy Memorandums (refer to Section 3.4).

#### **5.3.1 Order of Precedence**

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- TestAmerica QA/QC Policy Memorandum - Corporate
- Laboratory QA/QC Policy Memorandum
- Quality Assurance Manual
- Corporate SOPs and Policies

- Laboratory SOPs and Policies
- Other: Work Instructions (WI), memos, flow charts, etc.

#### **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 Precision**

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. The calculation of precision is described in Section 25.

##### **5.4.2 Accuracy**

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. The calculation of accuracy is described in Section 25.

### **5.4.3 Representativeness**

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 Comparability**

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, and by the degree to which approval from the US EPA or other pertinent regulatory agencies is obtained for any procedure for which significant modifications have been made.

### **5.4.5 Completeness**

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 Selectivity**

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), inter-element 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 Sensitivity**

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 prepares a Control Limit Report that summarizes the precision and accuracy acceptability limits for analyses performed at TestAmerica North Canton. This summary includes an effective date, is updated each time new limits are generated, and is located in QC Browser. The charts are stored as hard copies. 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, TestAmerica North Canton 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)]. TestAmerica North Canton 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 Group Leader 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. 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 QC Charts**

In-house limits for all QC data must be evaluated and compared to the limits published in the methods for applicable matrices. Method limits will be employed until sufficient QC data are acquired. A minimum of 20 to 30 data points are recommended to establish the in-house QC limits. Calculated results of the QC (LCS) samples are evaluated by comparing against control limits (3-sigma).

Control charts are used to develop control limits, trouble-shoot analytical problems, and, in conjunction with the non-conformance system, to monitor for trends. Program-specific data analysis requirements for control charts are followed as required for data generated under those programs. These additional requirements shall be documented in a QAPP or QAS.

## **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.

## SECTION 6

### DOCUMENT CONTROL (NELAC 5.4.3)

#### 6.1 OVERVIEW

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 at each laboratory Facility:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

The Corporate staff posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These are collectively termed “Official Documents” and encompass the Policies and Procedures that all facilities are required to employ. These official documents are only considered controlled when they are read on the company 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 official documents is found in Corporate SOP CW-Q-S-001, Corporate Document Control and Archiving.

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. Discussion on records control is described in Section 15.

The maintenance of purchasing data is discussed in Section 9.

The maintenance of sales and marketing contracts is discussed in Section 7.

#### 6.2 DOCUMENT APPROVAL AND ISSUE

The pertinent elements of a control system for each document include a unique name and number, the number of pages of the item, the effective date, revision number, and the laboratory

name. The QA Department is responsible for the maintenance of the system, and maintains the items electronically on the Laboratory public drive or the QA Department files.

Controlled documents are authorized by the QA Department and other management. In order to develop a new document, a staff member 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 as needed to those using it. Controlled documents shall be available at all locations where the operational activity described in the document is performed (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 24 months, and revised as appropriate. For procedures associated with DoD project work, SOPs and Policies are reviewed every 12 months. Changes to documents occur when a procedural change warrants a revision of the document.

### **6.3 PROCEDURES FOR DOCUMENT CONTROL POLICY**

For changes to the QA Manual, refer to SOPs NC-QA-002 and CW-Q-S-001. Uncontrolled copies must not be used within the laboratory. Previous revisions and back-up data are stored by the QA/QC Department. Electronic copies are stored on the Public server in the QA folder for the applicable revision.

For changes to SOPs, refer to SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-027, Preparation and Management of Standard Operating Procedures.

Forms, worksheets, work instructions, and information are organized by department in the QA office. Electronic versions are kept on a hard drive in the QA department; hard copies are kept in QA files. The procedure for the care of these documents is in SOP NC-QA-027.

### **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 as described in Section 15.

## SECTION 7

### REVIEW OF WORK REQUEST

#### 7.1 OVERVIEW

TestAmerica North Canton 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 TestAmerica'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 TestAmerica'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 (Percent 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 TestAmerica'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 review process 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 opportunity is forwarded to a Customer Service Manager (CSM) for review. The CSM contacts the appropriate Sales Executive (National Account Manager, Key Account Executive, Regional Account Executive, and/or Program Manager) to determine which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, reporting specifications, and available capacity to perform the work. The contract review process is outlined in SOP 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
- Laboratory Customer Service Manager
- Laboratory Operations Manager
- Laboratory and/or Corporate Technical Director
- Laboratory and/or Corporate Information Technology Managers/Directors
- Regional and/or National Account representatives
- Laboratory and/or Corporate Quality Managers
- 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.
- Based on the level of discount extended for the project, approval of the General Manager or Sales Director may also be required.

The Customer Service Manager or local Account Executive 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 backup will fulfill the review requirements.

The Legal & Contracts Director (or their designee) maintains copies of all signed contracts. The Laboratory Director also maintains an electronic copy of any contract signed at the local level.

### **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 Corporate Contracts Department and the applicable Account Executive. A copy of the contract will be filed electronically by the Laboratory Director. Quotes will be archived electronically in the laboratory quote module (TALs) or in the public shared drive if an off-TALs quote is submitted.

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 email records or 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, TestAmerica 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.

PM's are the direct 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. Project-specific changes made after samples are in-house are communicated through Change Order forms.

Programmatic and/or method changes are communicated via email transmittal and/or in meetings with the applicable Operations Managers. If the modification includes use of a non-standard method, or significant modification of a method, documentation of the modification is made in the case narrative of the applicable data report(s).

TestAmerica 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.

## SECTION 8

### SUBCONTRACTING OF TESTS (NELAC 5.4.5)

#### 8.1 OVERVIEW

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the corporate network. The phrase “work sharing” refers to internal transfers of samples between company 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 we must 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 the SOP 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.

For DOD projects, the subcontractor laboratories used must have an established and documented laboratory quality system that complies with DoD QSM requirements. The subcontractor laboratories are evaluated following the procedures outlined below and as seen in Figure 8-2. The subcontractor laboratory must receive project-specific approval from the DoD client before any samples are analyzed.

The QSM has five specific requirements for subcontracting:

1. Subcontractor laboratories must have an established laboratory quality system that complies with the QSM.
2. Subcontractor laboratories must be approved by the specific DoD Component laboratory approval process.
3. Subcontractor laboratories must demonstrate the ability to generate acceptable results from the analysis of PT samples, subject to availability, using each applicable method, in the specified matrix, and provide appropriate documentation to the DoD client.

4. Subcontractor laboratories must receive project-specific approval from the DoD client before any samples are analyzed.
5. Subcontractor laboratories are subject to project-specific, on-site assessments by the DoD client or their designated representatives.

Project Managers (PMs) or Customer Service Managers (CSM) 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 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 network 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 the company (in J.D.Edwards). A listing of all approved subcontracting laboratories and supporting documentation is available on the TestAmerica intranet site. Verify necessary accreditation for the requested tests prior to sending samples.
- 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 intra-company 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. Refer to SOP CA-C-S-001, Work Sharing Process.

When the potential subcontract laboratory has not been previously approved, CSMs 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. 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** The QA Manager must ensure that the Subcontracting Approval Form (Figure 8-2) has been completed and have supporting documentation on file prior to initiation of any work. A letter or e-mail is sent to the lab requesting the following information:

**8.2.1.1** If a lab is NELAC or A2LA-accredited:

**8.2.1.1.1** Copy of necessary certifications verifying the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.

**8.2.1.1.2** Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

**8.2.1.1.3** USDA soil permit, if available

**8.2.1.2** For laboratories accredited by other agencies with an auditing program

**8.2.1.2.1** Copy of necessary certifications verifying that the required approvals are current. Ensure all needed analytes are included; some may not be accreditable (if so, document). Certificate and scope of International Standard accreditation are required, when applicable.

**8.2.1.2.2** Insurance Certificate. This is required by the TestAmerica Chief Financial Officer

**8.2.1.2.3** USDA soil permit, if available\*\*

**8.2.1.2.4** Description of Ethics and Data Integrity Plan

**8.2.1.2.5** The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action

**8.2.1.2.6** State Audit with Corrective Action Response

**8.2.1.2.7** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)

**8.2.1.2.8** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are

grandfathered.)

- 8.2.1.2.9** DoD work includes additional requirements as described in Section 8.1 above.
- 8.2.1.3** For laboratories performing tests that are unaccredited or accredited by an agency without an audit program:
  - 8.2.1.3.1** A copy of their Quality Assurance Manual (controlled, if possible). Ensure data quality limits for relevant methods are acceptable and that training procedures are adequate.
  - 8.2.1.3.2** A copy of necessary certifications (if available) verifying the required approvals are current. Ensure all needed analytes are included. Some may not be accreditable (if so, document). Certificate and scope of international Standard accreditation are required, when applicable.
  - 8.2.1.3.3** Insurance Certificate. This is required by the Test America Chief Financial Officer.
  - 8.2.1.3.4** USDA soil permit, if available\*\*.
  - 8.2.1.3.5** Evidence of a current SOP per method. A copy of the first page and signature page of the SOP is acceptable. A Table of Contents including effective dates may also be acceptable. The SOP can be examined if an on-site audit is performed.
  - 8.2.1.3.6** Description of Ethics and Data Integrity Plan
  - 8.2.1.3.7** The most recent two sets of full proficiency testing (PT) results relevant to the analyses of interest and any associated corrective action.
  - 8.2.1.3.8** Example final report to confirm format is compliant and provides the necessary information. (Minimally, it must be determined the Batch QC results are included in the laboratory reports, and data is appropriately qualified.)
  - 8.2.1.3.9** Statement of Qualification (SOQ) or summary list of Technical Staff and Qualifications – position, education, and years of experience.
  - 8.2.1.3.10** DoD work includes additional requirements as described in Section 8.1 above.

\*\*USDA permit is required if soils less than three feet deep from New York, North Carolina, South Carolina, Georgia, Florida, Tennessee, Alabama, Mississippi, Louisiana, Arkansas, Texas, Oklahoma, New Mexico, Arizona, California, Hawaii, or outside the continental U.S. are to be analyzed. These samples require special shipping measures (check with the EHS Department). It may be necessary to heat-treat the samples before shipping if the subcontract laboratory does not have a USDA permit; however, some analytes/tests may be irrelevant after heat treatment.

**8.2.1.3.11** A copy of raw data associated with the first project is requested for internal review. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. If the QA Manager is unfamiliar with the analysis being performed, notify Corporate QA for guidance on the review. (It may need to be sent elsewhere for evaluation.) This requirement can be skipped if an on-site visit of the laboratory is planned. (This requirement is effective as of the effective date of this section. Laboratories worked with previously—minimum of six months—are grandfathered.)

**8.2.2** Once the information is received by the QA Manager, it is evaluated for acceptability 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 associated documentation and notify the Finance Group for J.D.Edwards.

**8.2.3** 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 to meet minimal standards. The company does not certify laboratories. The subcontractor is on our approved list, and can only be recommended to the extent that we would use them.

**8.2.4** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contract Department. Any problems identified will be brought to Corporate QA attention.

- Complaints shall be investigated. Documentation of the complaint, investigation, and corrective action will be maintained in the subcontractor file on the intranet site. Complaints must be posted using the Vendor Performance Report (Form CW-F-WI-009).
- Information must 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 network laboratories and Corporate QA 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 Directors.

### **8.3 OVERSIGHT AND REPORTING**

The CSM or 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 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 8-3) and the form is retained in the project folder. For network laboratories, certifications can be viewed on the company website.

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 the network.

The PM will communicate with the subcontracted laboratory to monitor the status of the analyses, facilitate successful execution of the work and ensure 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 laboratory EDD, i.e., imported, the report must explicitly indicate the specific lab that produced the data and identify the specific methods and samples.

**Note:** The results submitted by a network work sharing laboratory may be transferred electronically and the results reported by the network 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/Manager may waive the full qualification of a subcontractor process temporarily to meet emergency needs. In the event this provision is utilized, Corporate QA must be informed, and the QA Manager will be required to verify adequacy of proficiency scores and certifications. The laboratory must also request a copy of the raw data to support the analytical results for the first project submitted to the subcontract laboratory unless the laboratory has NELAC accreditation. The raw data is reviewed by the QA Manager and the PM to ensure that the results meet the client's needs. The QA Manager will request full documentation and qualify the subcontractor under the provisions above. The approval process should be completed within 30 calendar days of subcontracting.

**Figure 8-1.  
Example - Client-Approved Subcontractor Form**

**Client Information:**

Client Name & Account Number: \_\_\_\_\_

Client Contact: \_\_\_\_\_

Client Address: \_\_\_\_\_  
\_\_\_\_\_

**Project Information:** (Please choose all applicable.)

- ❖ Certification required:  State  NELAC  A2LA  Method \_\_\_\_\_  
 Target compound \_\_\_\_\_  Other \_\_\_\_\_  N/A  
❖ Required Turn around time (method provisional) \_\_\_\_\_

**Subcontractor's Information:**

Subcontractor's Name: \_\_\_\_\_

Subcontractor's Contact: \_\_\_\_\_

Subcontractor's Email: \_\_\_\_\_

Subcontractor's Address: \_\_\_\_\_  
\_\_\_\_\_

Subcontractor's Phone Number: \_\_\_\_\_

Analytical Test/Compound/Method to be subcontracted: \_\_\_\_\_  
\_\_\_\_\_

**Certification Statement:**

I hereby give *[Insert Lab Name]* permission to use the above noted subcontractor for the above noted testing procedures/methods. I realize that the above subcontractor will be held liable for the validity of the above mentioned testing procedures/methods. All subcontractors shall meet the requirements as spelled out in project information and will follow all analytical holding times and turn around times for analytical reports. The subcontract laboratory, and not TestAmerica, will be held liable for liquidated damages for delays in subcontracted analytical reports and/or electronic data deliverables.

\_\_\_\_\_  
Client Signature

\_\_\_\_\_  
Date

**Figure 8-2.**

**Example - Subcontracting Laboratory Approval Form (Initial / Renewal)**

**SUBCONTRACTING LABORATORY APPROVAL**

Reference: Section 8 – Quality Assurance Manual

Date: \_\_\_\_\_  
 Laboratory: \_\_\_\_\_  
 Address: \_\_\_\_\_  
 Contact and e-mail address: \_\_\_\_\_  
 Phone: Direct \_\_\_\_\_ Fax \_\_\_\_\_

Requested Item <sup>3</sup>	Date Received	Reviewed/ Accepted	Date
1. QA Manual <sup>3</sup>			
2. Copy of State Certification <sup>1</sup>			
3. State Audit with Corrective Action Response (or NELAC or A2LA Audit) <sup>3</sup>			
4. Most Recent (and relevant) 2 Sets of WP/WS Reports with Corrective Action Response <sup>1,3</sup>			
5. SOQ or Summary list of Technical Staff and Qualifications <sup>3</sup>			
6. SOPs for Methods to Be Loadshifted <sup>2,3</sup>			
7. USDA Soil Permit			
8. Insurance Certificate			
9. Sample Report <sup>3</sup>			
10. For DoD Work: Statement that Lab quality system complies with QSM			
11. For DoD Work: Approved by specific DoD Component laboratory approval process			
11. Description of Ethics Program <sup>3</sup>			

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 numbers 4 through 10 are not required.

On Site Audit Planned: YES NO If yes, Date Completed: \_\_\_\_\_ By Whom: \_\_\_\_\_

Comments: \_\_\_\_\_

Lab Acceptable for Subcontracting Work: YES NO Limitations: \_\_\_\_\_

QA Manager: \_\_\_\_\_ (Printed Name) Date: \_\_\_\_\_

Forwarded to Contract Coordinator by: \_\_\_\_\_ Date: \_\_\_\_\_

## SECTION 9

### PURCHASING SERVICES AND SUPPLIES (NELAC 5.4.6)

#### 9.1 OVERVIEW

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 the Controlled Purchases Procedure, CW-F-S-004. Only one quote is required where the item being purchased is a sole source product, Examples of sole source capital expenditures are laboratory test equipment, client specified purchases and building leases. A minimum of two quotes is required where the opportunity exists to source from more than one vendor. All documentation related to the purchase of capital items will be maintained in the individual CapEx files located in Corporate Purchasing. Data will be held in accordance with the record retention policy.

TestAmerica will enter into formal contracts with vendors when it is advantageous to do so. Contracts will be signed in accordance with the Authorization Matrix Policy, CW-F-P-002. Examples of items that are purchased through vendor contracts are laboratory instruments, consumables, copiers and office supplies. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. 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.

Non-capital expenditure items are purchased through the requisition and approval process in JD Edwards or through other TestAmerica authorized methods (approved web-sites, purchasing cards). Labs have the ability to select from the approved vendors in JD Edwards.

#### 9.2 GLASSWARE

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

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents must

meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP CA-Q-S-001.

### **9.3.1 Purchasing**

The nature of the analytical laboratory demands that all material used in any of the procedures is 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.

### **9.3.2 Receiving**

It is the responsibility of the Warehouse 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 kept on a backup disc located in the Wet Chemistry bullpen and 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 Specifications**

There are many different grades of analytical reagents available to the analyst. 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 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.

- An expiration date cannot 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 in the Standard Logbook in each laboratory group.

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 conductivity of less than 1 mmho/cm at 25°C. The conductivity is checked and recorded daily. If the water's conductivity is less than the specified limit, the Operations Manager 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) 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.

#### **9.3.4      Storage**

Reagent and chemical storage is important from the aspects of both integrity and safety. Light-sensitive reagents may be stored in brown-glass containers. Table 9-1 details specific storage instructions for reagents and chemicals. Section 22 discusses conditions for standard storage.

### **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 Operations Manager and/or the Laboratory Director/Manager. If they agree with the request the procedures outlined in Policy 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, it is given a short name, such as HP-20, added to the equipment list described in Section 21 that is maintained by the QA Department, and I.T. must be notified so it can be linked for backups. 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 (see 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 as specified in the laboratory's procedure for software verification. Software certificates supplied by the vendors are filed with the LIMS Administrator. 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 Department Managers or Operations Manager.

## **9.6 SUPPLIERS**

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). The level of control used in the selection process is dependent on the anticipated spend 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 (CW-F-WI-009).

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 New Vendor Procedure**

TestAmerica employees who wish to request the addition of a new vendor must complete a J.D. Edwards Vendor Add Request Form (CW-F-WI-007 – refer to Figure 9-2).

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.

**Table 9-1.**  
**Storage of Reagents and Chemicals**

<b>Chemical</b>	<b>Storage Requirements</b>
Concentrated Acids and Bases	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Bulk Dry Chemicals	Stored in the original containers at room temperature. All organic acids must be stored separately from inorganic acids. Acids should not be stored with bases.
Working Solutions containing Organic Compounds	Stored as per method recommendation/ requirement. They are generally stored refrigerated at $4^{\circ}\text{C} \pm 2^{\circ}\text{C}$ .
Working Solutions containing only Inorganics	Stored at room temperature; refrigeration is optional.
Flammable Solvents	Stored in solvent cabinets at room temperature.
Non-Flammable Solvents	Stored separately from the flammable solvents in cabinets at room temperature.

**Figure 9-2.**  
**Example – JD Edwards Vendor Add Request Form**



JD Edwards Vendor Add Request Form

Vendor name:	Lab location <u>and</u> individual making request:
Vendor address (remit to):	Vendor phone:
Vendor address (remit to):	Vendor fax:
Contact name:	Product / service provided:

**Reason for Vendor Addition: Check all reasons that apply**

<input type="checkbox"/> Cost Reduction	Estimated Annual Savings \$
<input type="checkbox"/> Replace Current Vendor	Reason?
	Vendor being Replaced?
<input type="checkbox"/> New Product / Service	Describe:
<input type="checkbox"/> ISO Approved ( <u>Required for Aerotech / P&amp;K only</u> )	

**Small Business:**

Does this vendor help us to meet our small business objectives: \_\_\_\_\_  
 If yes, which category: \_\_\_\_\_

**Personal and Ethical Considerations:**

Is there any personal conflict of interest with a TestAmerica employee and the vendor listed above? \_\_\_\_\_  
 Have ethical considerations been taken into account in your evaluation of this vendor? \_\_\_\_\_  
 \_\_\_\_\_

**Can this product be sourced from another TestAmerica facility?** \_\_\_\_\_

Please complete form and email to [NCPurchasing@testamericainc.com](mailto:NCPurchasing@testamericainc.com) or fax to (330) 966-9275.

I approve the addition of this vendor:

\_\_\_\_\_  
 Purchasing Manager - Patrick Eckman

\_\_\_\_\_  
 Corporate Controller - Leslie Bowers

Form No. CW-F-WI-007

## **SECTION 10**

### **SERVICE TO THE CLIENT (NELAC 5.4.7)**

#### **10.1 OVERVIEW**

TestAmerica North Canton 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 discussed in Section 5. The laboratory has procedures to ensure confidentiality to clients (Sections 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.

#### **10.2 SPECIAL SERVICES**

The laboratory's standard procedures for reporting data are described in Section 26. When requested the following special services are provided:

- The laboratory will provide the client or the client's representative reasonable access to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- The laboratory will work with client-specified third party data validators as specified in the client's contract.
- The laboratory will provide the client with all requested information pertaining to the analysis of their samples. An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

#### **10.3 CLIENT COMMUNICATION**

Customer Service Managers (CSMs) and Project Managers (PMs) are an important communication link to the clients. The lab shall inform its clients of any delays in project completion as well as any non-conformances in either sample receipt (refer to Section 24) or sample analysis. Project Management will maintain ongoing client communication throughout the entire client project.

A Technical Director, Operation Manager, or Lab Supervisors are available to discuss any technical questions or concerns that the client may have.

#### **10.4 REPORTING**

The laboratory will work with the client to produce any special communication reports required by the contract.

**10.5**      **CLIENT SURVEYS**

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service.

TestAmerica Sales and Marketing teams periodically develop lab and client-specific surveys to assess client satisfaction.

## **SECTION 11**

### **COMPLAINTS (NELAC 5.4.8)**

#### **11.1 OVERVIEW**

TestAmerica North Canton believes that effective client complaint handling processes have important business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that helps to continually improve processes and improving 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, 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 dealing with both external and internal complaints.

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 SOPs CORP-QA-0010, Nonconformance and Corrective Action System, and S-C-002, Complaint Handling and Service Recovery. It is the laboratory's goal to provide a satisfactory resolution to complaints in a timely and professional manner.

#### **11.2 EXTERNAL COMPLAINTS**

An employee that receives a complaint initiates the complaint resolution process and the documentation of the complaint.

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 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.

### **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 Information Technology (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)

## SECTION 12

### CONTROL OF NON-CONFORMING WORK (NELAC 5.4.9)

#### 12.1 OVERVIEW

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 13).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. Refer to SOP CORP-QA-0010, Nonconformance and Corrective Action System, for details of the nonconformance process.

#### 12.2 RESPONSIBILITIES AND AUTHORITIES

SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall, outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of the company'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/Manager, Operations Manager, Project Manager, or a member of the QA team may exceptionally 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 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 described in Section 13. This information may also need to be documented in logbooks and/or data review 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, QA Manager, Customer Service Manager, Operations Manager, I.T. Manager, H.R. Manager, and Technical Director. The reporting of issues involving alleged violations of the company's Data Integrity or Manual Integration procedures must be conveyed to an Ethics and Compliance Officer (ECO) and Quality Director within 24 hours.

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, COOs (East and West), General Managers and the Quality Directors (East and West) 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.

SOP CA-L-S-001 distinguishes between situations when it would be appropriate for the laboratory QA Manager and Laboratory Director/Manager (or his/her designee) 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 ECOs and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting (Section 13) in lieu of the data recall determination form contained in SOP 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 (Section 13).

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.

### **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 above.

Prior to suspension/restriction, confidentiality will be respected, and the problem and the required corrective and preventive action will be stated in writing and presented to the Laboratory Director/Manager.

The Laboratory Director/Manager 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 must 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/Manager 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, QA Manager, Supervisor) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, Director of Client Services, and Director of Sales and Marketing should 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 as described in Section 13.

## SECTION 13

### CORRECTIVE ACTION (NELAC 5.4.10)

#### 13.1 OVERVIEW

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 Nonconformance Memos (NCM).

#### 13.2 DEFINITIONS

- **Correction:** 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 are contained in the method-specific SOPs. 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 is not only a correction made to the immediate event, but a change in process, procedure or behavior that is required to eliminate the causes of an existing nonconformity, defect, or other undesirable situation in order to prevent recurrence.

#### 13.3 GENERAL

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 investigation.
- 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 (see more on client complaints in Section 11).

**13.3.1 Non-Conformance Memo (NCM)** - 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

#### **13.4 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.

##### **13.4.1 Cause Analysis**

- Upon discovery of a non-conformance event, the event must be defined and documented. An NCM must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 13-1 in SOP CORP-QA-0010, Nonconformance and Corrective Action System, 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, Lab Director, QA Manager, or designee is consulted.

##### **13.4.2 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 is used for this documentation.

##### **13.4.3 Monitoring of the Corrective Actions**

- The Department Manager/Supervisor and QA Manager is responsible to ensure that the corrective action taken was effective.
- Ineffective actions will be 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 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 NCMs 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 out-of-control situation and problems encountered in solving the situation.

#### **13.4.4 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. (Section 16 includes additional information regarding internal audit procedures.)
- 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.

#### **13.5 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 12 for information regarding the control of non-conforming work). The documentation of these procedures is through the use of an NCM.

Table 13-1 includes examples of general technical corrective actions. For specific criteria and corrective actions refer to the analytical methods or specific method SOPs.

Table 13-1 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, QAM Sections 20 and 21, and SOP CA-L-S-001, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall. All corrective actions are reviewed at a minimum monthly 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 a written NCM and appropriate corrective action (e.g., reanalysis) is taken and documented.

#### **13.6 BASIC CORRECTIONS**

When mistakes occur in records, each mistake shall be crossed-out, and not erased, deleted, made illegible, or otherwise 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.

**Table 13-1.  
 Example – General Corrective Action Procedures**

\* For Ohio VAP, method blank contamination must not exceed the RL if that analyte is a Contaminant of Concern for the project site.

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Alkalinity	* Method Blank	310.1 2320B	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration must be less than the reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	—	N/A
	Laboratory Control Sample	310.1 2320B	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples</p>	—	N/A
	Matrix Spike	310.1 2320B	<p><u>Total alkalinity:</u> 1 per batch of 20 samples</p>	—	N/A
	Matrix Spike Duplicate	310.1 2320B	<p><u>Total alkalinity:</u> 1 per batch of 20 samples</p>	—	N/A
	Duplicate	310.1 2320B	<p>For carbonate, bicarbonate, hydroxide, alkalinity only.</p> <p><u>Frequency:</u> 1 per batch of 10 samples <u>Criteria 310.1:</u> ≤ 20 % RPD<sup>(3)</sup></p> <p><u>Criteria 2320B:</u> ≤ 25 % RPD<sup>(3)</sup></p> <p><u>Corrective Action:</u> Flag data outside of limit.</p>	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Ammonia	* Method Blank	350.1 350.2 SM4500 NH-E,F	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	—	N/A
	Laboratory Control Sample	350.1 350.2 SM4500 NH-E,F	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within control limits, rerun all associated samples</p>	—	N/A
	Matrix Spike	350.1 350.2 SM4500 NH-E,F	<p><u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Ammonia (Cont'd)	Matrix Spike Duplicate	350.1 350.2 SM4500 NH-E,F	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Duplicate	350.1 350.2 SM4500 NH-E,F	N/A	—	N/A
Ammonia (TKN)	* Method Blank	351.2 351.3  SM4500 NO <sub>3</sub>	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Ammonia (TKN) (Cont'd)	Laboratory Control Sample	351.2 351.3  SM4500 NO <sub>3</sub>	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples</p>	—	N/A
	Matrix Spike	351.2 351.3  SM4500 NO <sub>3</sub>	<p><u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria:</u> Must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	—	N/A
	Matrix Spike Duplicate	351.2 351.3  SM4500 NO <sub>3</sub>	<p><u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria:</u> Must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	—	N/A
	Duplicate	351.2 351.3  SM4500 NO <sub>3</sub>	N/A	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
BOD	* Method Blank	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample  and Laboratory Control Sample Duplicate	405.1 SM5210B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	405.1 SM5210B	N/A	—	N/A
	Matrix Spike Duplicate	405.1 SM5210B	N/A	—	N/A
	Duplicate	405.1 SM5210B	N/A	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Bromide	* Method Blank	300.0A <sup>(5)</sup>	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample  and  Laboratory Control Sample Duplicate	300.0A <sup>(5)</sup>	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Matrix Spike	300.0A <sup>(5)</sup>	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with MS outside of limit
	Matrix Spike Duplicate	300.0A <sup>(5)</sup>	N/A	9056A	N/A
	Duplicate	300.0A <sup>(5)</sup>	N/A	9056A	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Chemical Oxygen Demand (COD)	* Method Blank	410.4 SM5220D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample and Laboratory Control Sample Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	410.4 SM5220D	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	410.4 SM5220D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	—	N/A
	Duplicate	410.4 SM5220D	N/A	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Chloride	* Method Blank	300.0A <sup>5</sup> 325.2 325.3	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A <sup>5</sup> 325.2 325.3	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A <sup>(b)</sup>	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Methods 9251 Corrective Action:</u> If not within laboratory control limits, rerun all associated samples <u>Method 9056/9253 Corrective Action:</u> Flag data associated with MS outside of limits

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Chloride (cont'd)	Matrix Spike Duplicate	325.2 325.3	<p><u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	9056A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits/&lt; 20 % RPD<sup>(3)</sup></p> <p><u>Corrective Action:</u> Flag data outside of limit</p> <p><u>Method 9056:</u> MSD is not applicable</p>
	Duplicate	300.0A <sup>(5)</sup> 325.2 325.3	N/A	9056A	N/A
Chlorine, Residual	* Method Blank	330.5	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration must be less than the reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	—	N/A
	Laboratory Control Sample	330.5	N/A	—	N/A
	Matrix Spike	330.5	N/A	—	N/A
	Matrix Spike Duplicate	330.5	N/A	—	N/A
	Duplicate	330.5	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> ≤ 20 % RPD<sup>(3)</sup></p> <p><u>Corrective Action:</u> Flag data outside of limit.</p>	—	Water

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Chromium (Cr <sup>+6</sup> )	* Method Blank	3500 Cr-D	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration must be less than the reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	7196A 3060A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>
	Laboratory Control Sample	3500 Cr-D	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples</p>	7196A 3060A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples prepped</p> <p><u>Criteria:</u> percent recovery for water must be within ± 15 % and for solids must be within ± 20%</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS</p>
	Matrix Spike	3500 Cr-D	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Must be within laboratory QC limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	3060A 7196A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Advisory limits are 75% - 125% recovery</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike</p>

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Chromium (Cr <sup>+6</sup> ) (Cont'd)	Matrix Spike Duplicate	3500 Cr-D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Advisory limits are 75% - 125% recovery  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	3500 Cr-D	N/A	7196A 3060A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> ≤ 20 % RPD <sup>(3)</sup> limit  <u>Corrective Action:</u> Flag data outside of limit.

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Conductivity, Specific	* Method Blank	120.1 SM2510B	N/A	9050A	Not Applicable
	Laboratory Control Sample	120.1 SM2510B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9050A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	120.1 SM2510B	N/A	9050A	N/A
	Matrix Spike Duplicate	120.1 SM2510B	N/A	9050A	N/A
	Duplicate	120.1 SM2510B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples  <u>Criteria:</u> ≤ 20 % RPD <sup>(3)</sup>  <u>Corrective Action:</u> Flag data outside of limit.	9050A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Cyanide (Amenable)	* Method Blank	335.1 SM4500C N-G	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.1 SM4500C N-G	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.1 SM4500C N-G	N/A	9012A	N/A
	Matrix Spike Duplicate	335.1 SM4500C N-G	N/A	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within lab control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	335.1 SM4500C N-G	N/A	9012A	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Cyanide (Total)	* Method Blank	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	335.2 335.3 335.4 4500-CN E	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9012A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Cyanide (Total) (cont'd)	Matrix Spike Duplicate	335.2 335.3 335.4 4500-CN E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed  Criteria: Percent recovery must be within laboratory control limits  Corrective Action: Flag data outside of limit	9012A	Frequency: 1 with each batch of samples processed not to exceed 20 samples  Criteria: Percent recovery must be within laboratory control limits  Corrective Action: Flag data associated with unacceptable Matrix Spike
	Duplicate	335.2 335.3 335.4	N/A	9012A	N/A
Flashpoint	* Method Blank	—	N/A	1010 ASTM D93-9	N/A
	Laboratory Control Sample	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike	—	N/A	1010 ASTM D93-9	N/A
	Matrix Spike Duplicate	—	N/A	1010 ASTM D93-9	N/A
	Duplicate	—	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD <sup>(3)</sup> must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate	1010 ASTM D93-9	Frequency: 1 per batch of ≤20 samples per matrix Criteria: RPD <sup>(3)</sup> must be ≤ 20% Corrective Action: Flag data associated with unacceptable Duplicate

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Fluoride	* Method Blank	300.0A <sup>(5)</sup> 340.2  SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable
	Laboratory Control Sample	300.0A <sup>(5)</sup> 340.2  SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within control limits, rerun all associated samples
	Laboratory Control Sample Duplicate	300.0A <sup>(5)</sup>  SM4500F-C,ISE	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within control limits, rerun all associated samples

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Flouride (Cont'd)	Matrix Spike	300.0A <sup>(5)</sup> 340.2  SM4500F- C,ISE	<u>Frequency:</u> 1 per 10 samples by IC  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with outside of limit
	Matrix Spike Duplicate	340.2  SM4500F- C,ISE	<u>Frequency:</u> 1 per 20 samples by IC  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit	340.2	N/A
	Duplicate	300.0A <sup>(5)</sup> 340.2  SM4500F- C,ISE	N/A	9056A	<u>Frequency:</u> 1 with each batch of samples processed  <u>Criteria:</u> RPD <sup>(3)</sup> must be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with duplicates outside of laboratory RPD <sup>(3)</sup> limits

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Hardness	* Method Blank	130.2 2340B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	130.2 2340B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	130.2 2340B	<u>Method 130.2:</u> 1 per 20 samples  <u>Method 2340B:</u>  <u>Frequency, Criteria, and Corrective Action:</u> See ICP Metals Method 200.7 Requirements	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Hardness (cont'd)	Matrix Spike Duplicate	130.2 2340B	<u>Method 130.2:</u> 1 per 20 samples  <u>Method 2340B:</u>  <u>Frequency, Criteria, and Corrective Action:</u> See ICP Metals Method 200.7 Requirements	—	N/A
	Duplicate	130.2 2340B	N/A	—	N/A
Iron, Ferrous & Ferric	* Method Blank	3500-Fe D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	3500-Fe D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Iron, Ferrous & Ferric (Cont'd)	Matrix Spike	3500-Fe D	<u>Frequency:</u> 1 every 10 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag associated data outside of limit	—	N/A
	Matrix Spike Duplicate	3500-Fe D	<u>Frequency:</u> 1 every 10 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag associated data outside of limit	—	N/A
	Duplicate	3500-Fe D	N/A	—	N/A
Nitrate	* Method Blank	300.0A <sup>(5)</sup> 353.2  SM4500-NO <sub>3</sub> -E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Nitrate (Cont'd)	Laboratory Control Sample	300.0A <sup>(5)</sup> 353.2  SM4500-NO <sub>3</sub> -E	Frequency: 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0A <sup>(5)</sup> 353.2  SM4500-NO <sub>3</sub> -E	Frequency: 1 per 10 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	Frequency: 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A <sup>(5)</sup> 353.2  SM4500-NO <sub>3</sub> -E	Frequency: 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Nitrate (cont'd)	Duplicate	300.0A <sup>(5)</sup> 353.2  SM4500- NO <sub>3</sub> -E	N/A	9056A	<u>Frequency:</u> 1 per 10 samples  <u>Criteria:</u> RPD <sup>(3)</sup> must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
Nitrite	* Method Blank	300.0A <sup>(5)</sup> 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0A <sup>(5)</sup> 354.1 353.2	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Nitrite (Cont'd)	Matrix Spike	300.0A <sup>(5)</sup> 354.1 353.2	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples
	Matrix Spike Duplicate	300.0A <sup>(5)</sup> 354.1 353.2	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A
	Duplicate	300.0A <sup>(5)</sup> 354.1 353.2	N/A	9056A	<u>Frequency:</u> 1 per 10 samples  <u>Criteria:</u> RPD <sup>(3)</sup> must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, flag all associated samples

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Nitrate-Nitrite	* Method Blank	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	—	N/A
	Laboratory Control Sample	353.2	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Matrix Spike Duplicate	353.2	Frequency: 1 per 20 samples, minimum of one per batch of samples processed Criteria: Percent recovery must be within laboratory control limits Corrective Action: Flag data outside of limit	—	N/A
	Duplicate	353.2	N/A	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
pH	* Method Blank	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Laboratory Control Sample	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Percent recovery must be within laboratory control limits Corrective Action: If not within laboratory control limits, rerun all associated samples	9040B 9045C	N/A
	Matrix Spike	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Matrix Spike Duplicate	150.1 SM4500H-B	N/A	9040B 9045C	N/A
	Duplicate	150.1 SM4500H-B	Frequency: 1 with each batch of samples processed not to exceed 10 samples per matrix Criteria: ≤ 20 % RPD <sup>(3)</sup> limit Corrective Action: Flag data outside of limit.	9040B 9045C	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Advisory limits are ≤ 20% RPD <sup>(3)</sup> Corrective Action: Flag data associated with unacceptable Duplicate
Phenolics	* Method Blank	420.1	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration must be less than the reporting limit Corrective Action: Rerun all samples associated with unacceptable blank	9065	Frequency: 1 with each batch of samples processed not to exceed 20 samples Criteria: Concentration less than reporting limit Corrective Action: Rerun all samples associated with unacceptable blank

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Phenolics (Cont'd)	Laboratory Control Sample	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag associated data
	Matrix Spike Duplicate	420.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag associated data	9065	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag associated data

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Phosphorus (Total and Ortho-phosphate)	* Method Blank	300.0 <sup>(4,5)</sup> 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	300.0 <sup>(4,5)</sup> 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples
	Matrix Spike	300.0 <sup>(4,5)</sup> 365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 10 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag associated data associated with MS outside of limits
	Matrix Spike Duplicate	365.3 365.1 SM4500P-E	<u>Frequency:</u> 1 per 20 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit	9056A	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Phosphorus (Total and Ortho-phosphate) (Cont'd)	Duplicate	300.0 <sup>(4,5)</sup> 365.3 365.1 SM4500P-E	N/A	9056A	<u>Frequency:</u> 1 with each batch of samples processed <u>Criteria:</u> RPD <sup>(3)</sup> must be within laboratory control limits <u>Corrective Action:</u> Flag data associated with duplicates outside of laboratory RPD <sup>(3)</sup> limits
Solids	* Method Blank	160.1 160.2 160.3 160.4 160.5 SM2540E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> If analyte level in method blank is $\geq$ RL for the analyte of interest in the sample, all associated samples with reportable levels of analyte are reprepared and reanalyzed.	—	N/A
	Laboratory Control Sample	160.1 160.2 160.3 160.4 160.5 SM2540E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, reprepare and rerun all associated samples	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Solids (Cont'd)	Matrix Spike	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Matrix Spike Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	N/A	—	N/A
	Duplicate	160.1 160.2 160.3 160.4 160.5 SM4500P-E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples  <u>Criteria:</u> Sample results should agree within 20% if both the sample and sample duplicate results are > 5 X RL  <u>Corrective Action:</u> Flag data outside of limit	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Sulfate	* Method Blank	300.0A <sup>(5)</sup> 375.4	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration must be less than the reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	9038 9056A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration must be less than the reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>
	Laboratory Control Sample	300.0A <sup>(5)</sup> 375.4	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples</p>	9038 9056A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Method 9038 Criteria:</u> Percent recovery must be within ± 15 %</p> <p><u>Method 9056 Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)</p>

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Sulfate (cont'd)	Matrix Spike	300.0A <sup>(5)</sup> 375.4	<u>Frequency:</u> 1 per 10 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9038 9056A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 10 samples ( 9038) or 20 samples (9056) <u>Method 9038 Criteria:</u> Limits are 75% - 125% recovery <u>Method 9056 Criteria:</u> Percent recovery must be within laboratory 0control limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	300.0A <sup>(5)</sup> 375.4	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Flag data outside of limit	9038 9056A	N/A
	Duplicate	300.0A <sup>(5)</sup> 375.4	N/A	9038 9056A	N/A
Sulfide	* Method Blank	376.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9030A	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration must be less than the reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Sulfide (Cont'd)	Laboratory Control Sample	376.1	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples</p>	9030A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag associated data</p>
	Matrix Spike	376.1	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	9030A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag associated data</p>
	Matrix Spike Duplicate	376.1	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag data outside of limit</p>	9030A	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Percent recovery must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag associated data Method 9034: Not Applicable</p>
	Duplicate	376.1	N/A	9030A	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Total Organic Carbon (TOC)	* Method Blank	415.1 SM5310D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	415.1 SM5310D	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	415.1 SM5310D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Total Organic Carbon (TOC) (cont'd)	Matrix Spike Duplicate	415.1 SM5310D	<u>Frequency:</u> 1 per 20 samples, minimum of one per batch of samples processed  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Flag data outside of limit	9060 Walkley-Black	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery must be within laboratory control limits <u>Corrective Action:</u> Reanalyze if sample remaining. If not, flag data associated with unacceptable Matrix Spike Duplicate
	Duplicate	415.1 SM5310D	N/A	9060 Walkley-Black	Not Applicable
Total Organic Halides (TOX)	* Method Blank	450.1 <sup>(5)</sup>	<u>Frequency:</u> 1 with each set of 8 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9020B	<u>Frequency:</u> Run in duplicate between each group of 8 analytical determinations <u>Criteria:</u> Concentration less than reporting limit or less than 2 X MDL or RL whichever is lower <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	450.1 <sup>(5)</sup>	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within laboratory control limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)	9020B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Percent recovery of analyte must be within 90-110% <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS (ICV)

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Total Organic Halides (TOX) (cont'd)	Matrix Spike	450.1 <sup>(5)</sup>	<p><u>Frequency</u>: 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria</u>: Must be within laboratory control limits</p> <p><u>Corrective Action</u>: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike</p>	9020B	<p><u>Frequency</u>: 1 per batch of 10 samples</p> <p><u>Criteria</u>: Must be within laboratory control limits</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike SOP NO. CORP-WC-0001</p>
	Matrix Spike Duplicate	450.1 <sup>(5)</sup>	<p><u>Frequency</u>: 1 per 20 samples, minimum of one per batch of samples processed</p> <p><u>Criteria</u>: Must be within laboratory control limits</p> <p><u>Corrective Action</u>: Reanalyze if sample remaining. If not, flag data with unacceptable Matrix Spike</p>	9020B	N/A
	Duplicate	450.1 <sup>(5)</sup>	N/A	9020B	N/A
Turbidity	* Method Blank	180.1	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: Concentration must be less than the reporting limit</p> <p><u>Corrective Action</u>: Rerun all samples associated with unacceptable blank</p>	—	N/A

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Turbidity (cont'd)	Laboratory Control Sample	180.1	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> If not within laboratory control limits, rerun all associated samples	—	N/A
	Matrix Spike	180.1	N/A	—	N/A
	Matrix Spike Duplicate	180.1	N/A	—	N/A
	Duplicate	180.1	<u>Frequency:</u> 1 per 10 samples  <u>Criteria:</u> Must be within laboratory QC limits  <u>Corrective Action:</u> Flag data outside of limit Not Applicable.	—	N/A
Mercury by CVAA & CVAFS	* Method Blank	200 series 1631E	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL, and samples are ND.	7000A series	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0003. Exception: If blank is above RL and samples are ND.

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Mercury by CVAA & CVAFS (Cont'd)	Laboratory Control Sample	200 series 1631E	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be within <math>\pm 20</math> %. 1631E is <math>\pm 23</math>%</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be within <math>\pm 20</math> %</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0003. Exception: If samples are ND, results are reported.</p>
	Matrix Spike	200 series 1631E	<p><u>Frequency:</u> with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125%</p> <p><u>Criteria:</u> Recovery should be within 75-125 %</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Recovery should be within 75-125 %</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MS. (See SOP CORP-MT-0003 for detailed corrective action procedure and for other QC procedures.)</p>
	Matrix Spike Duplicate	200 series 1631E	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples. 1631E frequency is 1 in 10 samples, 71-125% RPD 24%</p> <p><u>Criteria:</u> Recovery should be within 75-125 % , RPD<sup>(3)</sup> should be within 20%</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003</p>	7000A series	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Recovery should be within 75-125 % , RPD<sup>(3)</sup> should be within 20%</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable MSD SOP CORP-MT-0003</p>

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Mercury by CVAA & CVAFS (Cont'd)	Duplicate	200 series 1631E	N/A	7000A series	N/A
ICP Metals	* Method Blank	200.7 200.8	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results &lt;RL are also valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001</p>	6010B 6020	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit. Concentration less than reporting with the exception of lab common contaminants. Sample results &lt;RL are also valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank SOP CORP-MT-0001</p>
	Laboratory Control Sample	200.7 200.8	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be ± 85-115%. If LCS is biased high and samples are &lt;RL, the results are valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001</p>	6010B 6020	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery of analyte must be ± 20 %. If LCS is biased high and samples are &lt;RL, the results are valid.</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS SOP CORP-MT-0001. If samples are ND, results are reported.</p>

**INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
ICP Metals (Cont'd)	Matrix Spike	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Matrix Spike Duplicate	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD <sup>(3)</sup> must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Limits for percent recovery are 75-125%, RPD <sup>(3)</sup> must be within 20% <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike SOP CORP-MT-0001
	Duplicate	200.7 200.8	Not Applicable	6010B 6020	Not Applicable
	Serial Dilution	200.7 200.8	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001	6010B 6020	Frequency: 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> 10% difference. 10% difference only applied if sample results are >50 times IDL. <u>Corrective Action:</u> Flag data associated with unacceptable Serial Dilution SOP CORP-MT-0001

## INORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)

### *Footnotes*

---

- <sup>1</sup> National Pollutant Discharge Elimination System
- <sup>2</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- <sup>3</sup> RPD-Relative Percent Difference
- <sup>4</sup> Orthophosphate only
- <sup>5</sup> Method not listed in 40 CFR Part 136. Method 300.0 is a proposed 40CFR method. Specific state and/or region approval is required for NPDES.
- <sup>6</sup> Current promulgated method is a Guidance Method Only, SW-846, Final Update III, Rev.3, 12/96.

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Aromatic Volatiles by GC	* Method Blank	602	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: Concentration less than reporting limit</p> <p><u>Corrective Action</u>: Rerun all samples associated with unacceptable blank</p>	8021B	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: Concentration less than reporting limit</p> <p><u>Corrective Action</u>: Rerun all samples associated with unacceptable blank</p>
	Laboratory Control Sample	602	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery must be within acceptance limits given in method for each analyte</p> <p><u>Corrective Action</u>: Rerun all samples associated with unacceptable LCS</p>	8021B	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte must be within laboratory acceptance limits</p> <p><u>Corrective Action</u>: Rerun all samples associated with unacceptable LCS</p>
	Matrix Spike	602	<p><u>Frequency</u>: 1 per 10 samples from each site or 1 per month, whichever is more frequent</p> <p><u>Criteria</u>: percent recovery for each analyte should be within advisory limits given in method</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>	8021B	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte should be within laboratory acceptance limits</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Aromatic Volatiles by GC (Cont'd)	Matrix Spike Duplicate	602	N/A	8021B	<p><u>Frequency</u>: 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte should be within laboratory acceptance limits</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>
	Duplicate	602	N/A	8021B	N/A
	Surrogates	602	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS</u>: All surrogates must be within laboratory established control limits before sample analysis may proceed.</p> <p><u>Sample Criteria</u>: Re-extract samples or flag sample data not meeting surrogate criteria</p>	8021B	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS</u>: All surrogates must be within laboratory established control limits before sample analysis may proceed.</p> <p><u>Sample Criteria</u>: Reprepare and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	602	Optional: Internal standards are added to the method blank and all samples (QC included).	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Halogenated Volatiles by GC	* Method Blank	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> Concentration less than reporting limit <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	--	N/A	8021B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Halogenated Volatiles by GC (Cont'd)	Duplicate	--	N/A	8021B	N/A
	Surrogates	--	N/A	8021B	Surrogates spiked into method blank and all samples (QC included)  <u>Method Blank Criteria and LCS:</u> All surrogates must be within laboratory established control limits before sample analysis may proceed.  <u>Sample Criteria:</u> Reprep and reanalyze samples or flag sample data not meeting surrogate criteria.
	Internal Standards	--	N/A	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Herbicides	Laboratory Control Sample	615 <sup>3</sup>	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Percent recovery must be within acceptance limits given in method for each analyte  <u>Corrective Action:</u> Re-extract all samples associated with unacceptable LCS	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Percent recovery for each analyte must be within laboratory acceptance limits  <u>Corrective Action:</u> Re-extract and reanalyze all samples associated with unacceptable LCS
	Matrix Spike	615 <sup>3</sup>	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent  <u>Criteria:</u> Percent recovery for each analyte should be within advisory limits given in method  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Percent recovery for each analyte should be within laboratory acceptance limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	615 <sup>3</sup>	N/A	8151A	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte should be within laboratory control limits  <u>Corrective Action:</u> Flag data associated with unacceptable matrix spike sample

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Herbicides (cont'd)	Duplicate	615 <sup>3</sup>	N/A	8151A	N/A
	Surrogates	615 <sup>3</sup>	N/A	8151A	Surrogates spiked into method blank and all samples (QC included)  <u>Method Blank Criteria and LCS:</u> All surrogates must fall within laboratory established control limits before sample analysis may proceed.  <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	615 <sup>3</sup>	N/A	8151A	Optional
Pesticides/ PCBs	* Method Blank	608	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Reprepare and reanalyze all samples associated with unacceptable blank

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Pesticides/ PCBs (Cont'd)	Laboratory Control Sample	608	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	608	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent  <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8081A 8082	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Pesticides/ PCBs (cont'd)	Matrix Spike Duplicate	608	N/A	8081A 80882	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	608	N/A	8081A 8082	N/A
	Surrogates	608	Not specified in method	8081A 8082	Surrogates spiked into method blank and all samples (QC included)  <u>Method Blank Criteria and LCS:</u> Results must fall within laboratory established control limits  <u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
Petroleum Hydrocarbons	* Method Blank	1664A	<u>Frequency:</u> 1 with each preparation batch  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	9071B	<u>Frequency:</u> 1 with each preparation batch  <u>Criteria:</u> Concentration must be less than the reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Petroleum Hydrocarbons (Cont'd)	Laboratory Control Sample	1664A	<u>Frequency:</u> 1 with each analytical batch  <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	9071B	<u>Frequency:</u> 1 with each analytical batch  <u>Criteria:</u> Waters - See limits in SOP, NC-WC-0084 Soils - Percent recovery must be within laboratory control limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	1664A	<u>Frequency:</u> 1 with every 10 samples per site  <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084  <u>Corrective Action:</u> See SOP, NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site  <u>Criteria:</u> See percent recovery limits in SOP, NC-WC-0084  <u>Corrective Action:</u> See SOP, NC-WC-0084
	Matrix Spike Duplicate	1664A	<u>Frequency:</u> 1 with every 20 samples per site  <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084  <u>Corrective Action:</u> See NC-WC-0084	9071B	<u>Frequency:</u> 1 with every 10 samples per site  <u>Criteria:</u> See percent recovery and RPD limits in SOP, NC-WC-0084  <u>Corrective Action:</u> See NC-WC-0084
	Duplicate	1664A	N/A	9071B	N/A

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Purgeable Halocarbons by GC	* Method Blank	601	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> Concentration less than reporting limit  <u>Corrective Action:</u> Rerun all samples associated with unacceptable blank
	Laboratory Control Sample	601	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery must be within acceptance limits given in method for each analyte  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits  <u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS
	Matrix Spike	601	<u>Frequency:</u> 1 per 10 samples from each site or 1 per month, whichever is more frequent <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8021B	<u>Frequency:</u> 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES<sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846)<sup>2</sup></b>
Purgeable Halocarbons by GC (Cont'd)	Matrix Spike Duplicate	601	N/A	8021B	<u>Frequency</u> : 1 with each extraction batch of samples not to exceed 20 samples <u>Criteria</u> : percent recovery for each analyte should be within laboratory acceptance limits <u>Corrective Action</u> : Flag data associated with unacceptable Matrix Spike
	Duplicate	601	N/A	8021B	N/A
	Surrogates	601	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract samples or flag sample data not meeting surrogate criteria	8021B	Surrogates spiked into method blank and all samples (QC included) <u>Method Blank Criteria and LCS</u> : All surrogates must be within laboratory established control limits before sample analysis may proceed. <u>Sample Criteria</u> : Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria
	Internal Standards	601	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.	8021B	Optional: Internal standards are added to the method blank and all samples (QC included). If used, same compounds as used for surrogates may be appropriate.

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Semivolatiles	Matrix Spike	625	<p><u>Frequency</u>: 1 with each extraction batch of samples not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte should be within advisory limits given in method</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>	8270C	<p><u>Frequency</u>: 1 with each extraction batch of samples not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte should be within laboratory acceptance limits</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>
	Matrix Spike Duplicate	625	N/A	8270C	<p><u>Frequency</u>: 1 with each extraction batch of samples not to exceed 20 samples</p> <p><u>Criteria</u>: percent recovery for each analyte should be within laboratory acceptance limits</p> <p><u>Corrective Action</u>: Flag data associated with unacceptable Matrix Spike</p>

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Semivolatiles (Cont'd)	Duplicate	625	N/A	8270C	N/A
	Surrogates	625	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank and LCS Criteria:</u>            All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery.</p> <p><u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria</p>	8270C	<p>Surrogates spiked into method blank and all samples (QC included)</p> <p><u>Method Blank and LCS Criteria:</u>            All surrogates must be in control before sample analysis may proceed. One surrogate per fraction may exceed control limits if greater than 10% recovery.</p> <p><u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	625	<p><u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included)</p> <p><u>Criteria:</u> All internal standard recoveries must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements</p>	8270C	<p>Internal Standards are added to all samples (QC samples included). Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard.</p> <p>The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed <math>\pm 0.5</math> minutes from mid level initial calibration standard IS RT.</p>

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

Analysis	QC Sample	Method	NPDES <sup>1</sup>	Method	RCRA (SW846) <sup>2</sup>
Volatiles by GC/MS	* Method Blank	624	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>	8260B	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> Concentration less than reporting limit</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable blank</p>
	Laboratory Control Sample	624	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method</p> <p><u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike</p>	8260B	<p><u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples</p> <p><u>Criteria:</u> percent recovery for each analyte must be within laboratory acceptance limits</p> <p><u>Corrective Action:</u> Rerun all samples associated with unacceptable LCS</p>

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Volatiles by GC/MS (Cont'd)	Matrix Spike	624	<u>Frequency:</u> 1 per ≤ 20 samples from each site or 1 per month, whichever is more frequent  <u>Criteria:</u> percent recovery for each analyte should be within advisory limits given in method  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Matrix Spike Duplicate	624	N/A	8260B	<u>Frequency:</u> 1 with each batch of samples processed not to exceed 20 samples  <u>Criteria:</u> percent recovery for each analyte should be within laboratory acceptance limits  <u>Corrective Action:</u> Flag data associated with unacceptable Matrix Spike
	Duplicate	624	N/A	8260B	N/A

**ORGANIC LABORATORY QUALITY CONTROL SAMPLES (Cont'd)**

<b>Analysis</b>	<b>QC Sample</b>	<b>Method</b>	<b>NPDES <sup>1</sup></b>	<b>Method</b>	<b>RCRA (SW846) <sup>2</sup></b>
Volatiles by GC/MS (cont'd)	Surrogates	624	<p>Surrogates spiked into Method Blank and all samples (QC included)</p> <p><u>Method Blank Criteria:</u> All surrogates must be in control before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Re-extract samples or flag sample data not meeting surrogate criteria</p>	8260B	<p>Surrogates spiked into Method Blank and all samples (QC included)</p> <p><u>Method Blank Criteria and LCS:</u> All surrogates must be in control before sample analysis may proceed.</p> <p><u>Sample Criteria:</u> Re-extract and reanalyze samples or flag sample data not meeting surrogate criteria</p>
	Internal Standards	624	<p><u>Frequency:</u> Internal standards spiked into method blank and all samples (QC included)</p> <p><u>Criteria:</u> All internal standard recoveries must be within laboratory control limits</p> <p><u>Corrective Action:</u> Flag sample data not meeting internal standard recovery requirements</p>	8260B	<p>Internal Standards are added to all samples (QC samples included).</p> <p>Internal standard area of daily standard must be within 50% to 200% of the response in the mid level of the initial calibration standard.</p> <p>The retention time (RT) for any internal standard (IS) in the continuing calibration must not exceed <math>\pm 0.5</math> minutes from mid level initial calibration standard IS RT.</p>

***Footnotes***

---

- <sup>1</sup> National Pollutant Discharge Elimination System
- <sup>2</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996)
- <sup>3</sup> Method not listed in 40 CFR Part 136

## SECTION 14.0

### PREVENTIVE ACTION (NELAC 5.4.11)

#### 14.1 OVERVIEW

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 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 our TestAmerica North Canton commitment to the Quality Assurance (QA) 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 Quality Assurance 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 (Section 13) 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:

- Identification of an opportunity for preventive action.
- Process 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.
- Close-Out 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, management review, and the Management of Change process (see below).

**Note:** There may be varying levels of formality and documentation during the preventive action process due to the simplicity/complexity of the action taken.

**14.1.2** 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.

## **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 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, Key Personnel Changes, Laboratory Information Management System (LIMS) changes. This process is discussed in further detail in SOP CA-Q-S-003, Management of Change.

## SECTION 15.0

### CONTROL OF RECORDS (NELAC 5.4.12)

TestAmerica North Canton 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 as described in SOP NC-QA-0019, Records Information Management.

#### 15.1 OVERVIEW

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 are maintained by the Quality Assurance (QA) Manager in a database which is backed up as part of the regular network 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 the Records Manager.

**Table 15-1. Record and Retention Schedule**

Type of Record	Retention	Disposition
<b>General Laboratory Documents</b>		
Instrument output	5 yrs from project completion	Shred or burn
Quality control data	5 yrs from project completion	Shred or burn
Field sample data	5 yrs from project completion	Shred or burn
Final analytical reports	5 yrs from project completion	Shred or burn
Instrument logbooks	5 yrs from last entry	Shred or burn
Equipment monitoring & maintenance records	5 yrs from last entry	Shred or burn
Instrument calibration records	5 yrs from last entry	Shred or burn
Standard preparation logs	5 yrs from last entry	Shred or burn
Standards certificates	5 yrs from last entry	Shred or burn
Measurement & test equipment logs (e.g., refrig., balances, etc.)	5 yrs from last entry	Shred or burn
Method & instrument validation records	5 yrs from last entry	Shred or burn
Instrument manuals	Retain until superseded	Trash
Project management files	5 yrs from date of archival	Shred or burn
Quotes & proposals	2 yrs from date of expiration	Shred or burn

Type of Record	Retention	Disposition
LQM, policies, & SOPs	5 yrs from date of archiving	Shred or burn
Analyst demonstrations of proficiency	5 yrs from date of archival	Shred or burn
Quality assurance audits	5 yrs from last entry	Shred or burn
Certifications & approvals	5 yrs from last entry	Shred or burn
Employee signature list	5 yrs from date of archival	Shred or burn
MDL Studies	5 yrs from last entry	Shred or burn
Performance testing studies	5 yrs from last entry	Shred or burn
QA reports to management	5 yrs from last entry	Shred or burn
Quality control charts	5 yrs from last entry	Shred or burn
<b>Environment, Health and Safety Records</b>		
Medical records	Retain while active & 30 yrs from last entry	Shred or burn
Employee exposure & monitoring records	Retain while active & 30 yrs from last entry	Shred or burn
Workers compensation files & first report of injury	Retain while active & 30 yrs from last entry	Shred or burn
Accident logs (OSHA Form 200)	5 yrs from last entry	Shred or burn
Accident reports	5 yrs from last entry	Shred or burn
Environmental permits	5 yrs from last entry	Shred or burn
Environmental management, e.g., discharge reports	5 yrs from last entry	Shred or burn
Health & safety audits	5 yrs from last entry	Shred or burn
Chemical Hygiene Plan	5yrs from archival	
Safety Inspections	5 yrs from last entry	Shred or burn
TLD exposure records	5 yrs from last entry	Shred or burn
EH&S training	5 yrs from last entry	Shred or burn
<b>Accounting</b>	See Accounting and Controls Procedures Manual	
<b>Administrative</b>		
Personnel records (not including medical or disability records)	7 years from last entry	Shred or burn

All records are legible and stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. 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. Policy CW-L-P-001, Record Retention, provides additional information on record retention requirements.

**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 15-3 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.

**Table 15-2. Special Record Retention Requirements**

Program	Retention Requirement
Ohio – Drinking Water	10 years
Michigan Department of Environmental Quality – all environmental data	10 years
Navy Facilities Engineering Service Center (NFESC)	10 years
OSHA - 40 CFR Part 1910	30 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement and others as negotiated.
Ohio Voluntary Action Program	10 years

**15.1.2** All records are held secure and in confidence. Records maintained at the laboratory are located in the Records Storage area of the Warehouse. Records archived off-site are stored in a secure location where a record is maintained of any entry into the storage facility. Logs are maintained in each storage box to note removal and return of records.

**15.1.3** 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 hardcopy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, see Section 20.12.1, Computer and Electronic Data Related Requirements, for more information.

**15.1.4** 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 two 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 copy of the Chain-of-Custody is stored with the invoice and the Work Order sheet generated by 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. SOP NC-QA-0019, Records Information Management, outlines this procedure. 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 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 which was scanned.
- Also refer to Section 20.13.1, "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 (refer to Section 15.1). 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 sample preparation, performance of each analysis and checking of results.

**15.2.2** Observations, data and calculations are recorded at the time they are made and are identifiable to the specific task.

**15.2.3** Changes to hardcopy records shall follow the procedures outlined in Sections 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 (previous discussions relate where most of this information is maintained – specifics may be added below):

- Laboratory sample ID code
- Date of analysis and time of analysis is 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. Instrument logs may be in electronic format.
- analysis type
- all manual calculations and manual integrations
- analyst or operator initials/signature
- sample preparation
- 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
- 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**

Sample handling and tracking is discussed in Section 24. 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 hardcopy form (see 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 to the accrediting body 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 hardcopy, write-protected backup copies, or an electronic audit trail controlling access.

**15.5.4** TestAmerica North Canton has a record management system for control of instrument logbooks, standards logbooks, and records for data reduction, validation, storage, and reporting. Benchsheets are filed sequentially per method.

**15.5.5** Records are considered archived when moved to Records Storage. Access to archived hard-copy information is documented with an access log and in/out records is used in archived boxes to note data that is removed and returned. 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.

**15.5.6** In the event that the laboratory transfers ownership or goes out of business, TestAmerica North Canton 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 five years of such action.

#### **15.5.7 Records Disposal**

**15.5.7.1** Records are removed from the archive and disposed after five 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.

**15.5.7.2** Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

**15.5.7.3** If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required. Refer to Policy CW-L-P-001, Records Retention.

## SECTION 16

### AUDITS (NELAC 5.4.13)

#### 16.1 OVERVIEW

Audits measure laboratory performance and insure compliance with accreditation/certification and project requirements. Audits specifically provide management with an on-going assessment of the quality of results produced by the laboratory, including how well the policies and procedures of the QA system and the Ethics and Data Integrity Program are being executed. They are also instrumental in identifying areas where improvement in the QA system will increase the reliability of data. There are two principle types of audits: Internal and External. Internal audits are performed by laboratory or corporate personnel. External audits are conducted by regulators, clients or third-party auditing firms. In either case, the assessment to program requirements is the focus.

**Table 16-1. Audit Types and Frequency**

Internal Audits	Description	Performed by	Frequency
	Analyst & Method Compliance	QA Department or Designee	- 100% of all methods over a two year period. - 100% of all analysts annually.
	Instrument	QA Department or Designee	100% of all organic instruments and any inorganic chromatography instruments. Annually.
	Work Order/ Final Report	QA Department or Designee	- 1 complete report each month.
	Support Systems	QA Department or Designee	- Annual for entire labs support departments & equipment (e.g., thermometers, balances), can be divided into sub-sections over the course of the year.
	Performance Audits (Double-Blind PTs)	Corporate QA, Laboratory QA Department or Designee	- As needed.
	Special	QA Department or Designee	- As Needed
External Audits	Description	Performed by	Frequency
	Program / Method Compliance	Regulatory Agencies, Clients, accreditation organizations	- As required by program and/or clients needs
	Performance Audits	Provided by a third party.	- As required by a client or regulatory agency. Generally provided semi-annually through the analysis of PT samples.

#### 16.2 INTERNAL AUDITS

Annually, the laboratory prepares a schedule of internal audits to be performed throughout the year. As previously stated, these audits verify and monitor that operations continue to comply with the requirements of the laboratory's QA Manual and the Corporate Ethics Program. A

schedule of internal audits is maintained by the QA Manager in the *Internal Audit Workbook*. An example can be found in Figure 16-1.

It is the responsibility of the QA Manager to plan and organize audits in consideration of the laboratory workload and the department personnel schedules so that all pertinent personnel and operations are thoroughly reviewed. When designees (other than QA Department personnel and approved by the QA Manager) perform audits, the QA Manager shall ensure that these persons do not audit their own activities, except when it can be demonstrated that an effective audit will be carried out. In general, the auditor:

- Is neither the person responsible for the process being audited nor the immediate supervisor of the person responsible for the project/process.
- Is free of any conflicts of interest.
- Is free from bias and influences that could affect objectivity.

Laboratory personnel (e.g., supervisors and analysts) may assist with both method and support system audits as long as the items listed in the above paragraph are observed. These audits are conducted according to defined criteria listed in the checklists of the *Internal Audit Workbook*. These personnel must be approved by the QA Manager; and must complete the audit checklists in their entirety. This process introduces analyst experience and insight into the laboratory's auditing program.

The auditor must review the previous audit report and identify all items for verification of corrective actions. A primary focus will be dedicated to the ability of the laboratory to correct root-cause deficiencies and that the corrective action has been implemented and sustained as documented.

### **16.2.1 Systems**

An annual Systems audit is required to ensure compliance to analytical methods and SOPs, laboratory Data Integrity and Ethics Policies, NELAC quality systems, client, and State requirements. This audit is performed in portions throughout the year through method, analyst, instrument, Work Order/Final Report, and support system audits. Audits are documented and reported to management within one week of their performance. Systems audits cover all departments of the facility--both operational and support. The multiple audits are compiled into one systems audit package at the end of the year (*Internal Audit Workbook*).

#### **16.2.1.1 Method, Analyst, Instrument and Work Order/Final Report Audits**

Procedures for the method compliance, analyst, instrument and work order/final report audits are incorporated by reference to SOP CA-Q-S-004, Method Compliance and Data Authenticity Audits. These audits are not mutually exclusive. For example, the performance of a method audit will also cover multiple analysts and instruments. The laboratory's goal is to annually review all analysts and instruments as described in SOP CA-Q-S-004. The laboratory will also audit all methods within a two-year time period, and audit a minimum of one Work Order/Final Report from receiving through reporting on a monthly basis.

### **16.2.1.2 Support Systems**

Support system audits are performed to ensure that all departments & ancillary equipment are operating according to prescribed criteria. Support system audits include the review of both non-analytical and operational departments. Support equipment audits, e.g., metrology items, include the review of balance calibrations, weight calibrations; water quality testing, etc.. Non-analytical may include sample receiving and bottle preparation. These types of support audits ensure that the operations are being performed to support ethical data as well as ensuring the accuracy & precision of the utilized equipment.

These audits can be performed in portions throughout the year or in one scheduled session. However, the audit schedule must document that these aspects are reviewed annually. Many of the metrology systems are considered to be surveillance activities that can be monitored by QA personnel or delegated to specified department personnel. These surveillance activities are performed on a semi-annual basis unless issues warrant a greater frequency or previous audits continually showing no deficiencies allow the frequency to be reduced to once a year.

An example audit checklist can be found in Figure 16-2. Instructions for reporting findings are included in the *Internal Audit Workbook*. In general, findings are reported to management within 1 week of the audit and a response is due from management within 30 days.

### **16.2.2 Performance Audits**

Corporate QA may arrange for double blind PT studies to be performed in the laboratories. Results are given to Management and Corrective actions of any findings are coordinated at each facility by the QA Managers and Laboratory Directors/Managers. These studies are performed on an as needed basis. They may be performed when concerns are raised regarding the performance of a particular method in specific laboratories, periodically to evaluate methods that may not normally be covered in the external PT program or may be used in the process of developing best practices. The local QA Manager may also arrange for PT studies on an as needed basis (refer to Section 16.3.2 for additional information on Performance Audits).

### **16.2.3 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.

## **16.3 EXTERNAL AUDITS**

TestAmerica facilities are routinely audited by clients and external regulatory authorities. External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is the TestAmerica 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. The 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. This time frame is generally 30 days.

Be aware that NELAC requires that the audit response report be acceptable to the primary accrediting authority after the second submittal. The lab shall have accreditation revoked for all or any portion of its scope of an accreditation for any or all fields of testing, a method, or analyte within a field of testing if it is not corrected.

TestAmerica North Canton cooperates with clients and their representatives to monitor laboratory 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.

### **16.3.1 Confidential Business Information (CBI) Considerations**

During on-site audits, on-site 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.

### **16.3.2 Performance Audits**

The laboratory is involved in performance audits conducted as directed by state agencies through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies—water pollution and soil studies.

- It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Further, 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.
- PTs generally do not have holding times associated with them. In the absence of any holding time requirement, it is recommended that the holding time begin when the PT sample is prepared according to the manufacturer's instructions. Holding times should apply to full volume PT samples only if the provider gives a meaningful "sampling date". If this is not provided, it is recommended that the date/time of opening of the full volume sample be considered the beginning of holding time.
- Login will obtain the COC information from the documentation provided with the PTs with review by QA or other designated staff.

- Vials will be prepared as required in the instruction set provided with the samples. After preparation to full volume the sample may be spiked, digested, concentrated, etc., as would be done for any normal sample requiring similar analysis.
- PT samples will not undergo multiple preps, multiple runs, multiple methods (unless being used to evaluate multiple methods), multiple dilutions, UNLESS this is what would be done to a normal client sample. An example of this is if a client requests, as PT clients do, that we split VOA coeluters, then dual analysis IS normal practice.
- The type, composition, concentration and frequency of quality control samples analyzed with the PT samples shall be the same as with routine environmental samples.
- Instructions may be included in the laboratory SOPs on how low-level samples are analyzed, including concentration of the sample or adjustment of the normality of titrant. When a PT sample falls below the range of the routine analytical method, the low-level procedure may be used. Results below the routine reporting limit are reviewed against PT instructions and analyte PT reporting limit values.
- No special reviews shall be performed by operation and QA, UNLESS this is what would be done to a normal client sample. To the degree that special report forms or login procedures are required by the PT supplier, it is reasonable that the laboratory WOULD apply special review procedures, as would be done for any client requesting unusual reporting or login processes.
- 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.

#### **16.4      AUDIT FINDINGS**

Internal or External Audit findings should be documented using the corrective action process. The laboratory is expected to prepare a response to audit findings within 30 days of receipt of an audit report unless the report specifies a different time frame. The response may include action plans that could not be completed within the 30-day timeframe. In these instances, a completion date must be set and agreed to by Operations Management and the QA Manager.

Responsibility for developing and implementing corrective actions to findings is the responsibility of the Group Leader where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report.

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 must take timely corrective action, and must 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.

The procedures must be in accordance to SOP CA-L-S-001, Internal Investigations of Data Discrepancies and Determination of Data Recall.

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.

Figure 16-1.

Example - Internal Audit Workbook

						<b>TestAmerica &lt;Name&gt;</b> Last Updated: 9/10/2007
THE LEADER IN ENVIRONMENTAL TESTING						Workbook Instruction No. CA-Q-WI-011
<b>Internal Audit Workbook</b> <b>Summary Page</b>						
<b>Note:</b> Click on the <a href="#">(Summary Page)</a> to located on each audit sheet to return to this page.						
<b>* The lab may choose to audit these areas with each method/analyst/instrument audit. The auditor must document on the checklist that this item is audited as part of the &lt;defined&gt; audit.</b>						
Area Audited	Audit Type	Audit Cycle	Scheduled	Date Audited	Date Closed	Comments
(Click on the Area to get to that Spreadsheet)						
1 Balances	System	6 mo				
2 Temperature Logs/Thermometers	System	6 mo				
3 Sample Storage and Disposal	System	1 yr				
4 Maintenance Logs *	System	6 mo				
5 Volatile Storage Blanks	System	6 mo				
6 Lab Water Quality Testing	System	6 mo				
7 Sample Log In	System	1 yr				
8 Shipping Procedures	System	1 yr				
9 Computer Operations (LIMS)	System	1 yr				Pending Corp. IT Policies
10 SOP & Document Distribution System	System	1 yr				
11 Archiving Electronic & Paper Records	System	1 yr				Pending Corp. IT Policies
12 Statistical Process Control	System	1 yr				
13 Data Review System	System	1 yr				
14 Final Report Generation	System	1 yr				
15 Standards/Reagents *	System	6 mo				
16 Manual Integration *	System	1 yr				
17 Corrective Action System	System	1 yr				
18 Training Records	System	6 mo				
19 MDLs	System	1 yr				
20 SOPs - Prep/Review/Update Process	System	1 yr				
21 Purchasing/Procurement	System	1 yr				
22 Eppendorf/Diluter/Dispenser Calibration Check	System	6 mo				
23 Subcontract Lab Approval	System	1 yr				
24 Customer Complaint System	System	1 yr				
25 Methods	Method	2 yr				
.....	.....	.....				
.....	.....	.....				
.....	.....	.....				

Figure 16-2.

Example – Internal Audit System Checklist: Corrective Actions



(Summary Page)

TestAmerica <Location>

INTERNAL AUDIT - Corrective Actions

[ Printed Name(s) or Date(s) ]

Area Audited:

Auditor:

Date:

Persons Contacted During Audit:

Date Reported to Department Manager:

Reported To:

Date Reported to Lab Director/Manager:

Reported To:

Date Response Due:

Response Received and Accepted by QA Manager:

Associated Corrective Action Report Number(s):

Scheduled Follow-up:

Item	Requirement	Ref.	Y	N	NA	Evidence/Comments	Follow Up
1	Does the laboratory have a corrective action program in place?	5.4.10.1					
2	Does the laboratory have a current corrective action SOP or is this information in the QA Manual?	5.4.10.1					
3	Do all laboratory personnel have documented training and access to initiate corrective actions?	5.4.10.1					
4	Are causes clearly identified by department, staff name, scope of issue (how many reports affected)?	5.4.10.6					
5	Is a root cause for the issue identified?	5.4.10.2					
6	Is a corrective action (plan) clearly described?						
7	Was the corrective action fully implemented?						
8	Is documentation (if applicable) completed as specified by the corrective action (training, revised SOP, etc)						
9	Has a follow-up assessment been conducted to verify the corrective action was successful?						
10	Are corrective actions reviewed on a regular basis by management?	5.4.10.6a 5					
11	Is there a defined distribution flow for corrective action notification, review, closure, and follow-up?	5.4.10.6a					
12	Are non-conformances reviewed on a regular basis and used, if necessary, to initiate root cause corrective actions?						
13	Does the lab have a documented procedure for QC corrective action (i.e., documented within each method / parameter SOP or in the QA Manual)?	4.10.1					
14	<b>Verify Corrective Actions from previous systems audits. List Items:</b>						
15							
16							
17							

Auditor Signature: \_\_\_\_\_

Primary Reference(s): Corporate SOP CA-Q-S-002, Acceptable Manual Integration Practices  
 NELAC Standard, June 2003  
 DoD Quality Systems Manual, Version 3, January 2006  
 EPA Manual for the Certification of Laboratories Analyzing Drinking Water

## 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/Manager for review and comments. The final report shall be submitted to the Laboratory Director 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. At a minimum, the report content will contain the items listed below. During the course of the year, the Laboratory Director/Manager, General Manager or Corporate QA may request that additional information be added to the report.

The TestAmerica QA Report template is comprised of a discussion of three key QA issues facing the laboratory and ten specific sections (Figure 17-1):

- **Metrics:** Describe actions or improvement activities underway to address any outlying quality metrics that have been reported in the monthly Quality System Metrics Table.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).
- **SOPs:** Report SOPs that have been finalized and report status of any outstanding SOP reviews.
- **Corrective Actions:** Describe highlights and the most frequent cause for report revisions and corrective/preventive action measures underway. Include a discussion of any recalls handled at the lab level as per Section 6.2.2 in the Investigation/Recall SOP, CA-L-S-001. Include a section for client feedback and complaints. Include both positive and negative feedback. Describe the most serious client complaints and resolutions in progress.
- **MDLs and Control Limits:** Report which MDLs/ MDL verifications are due. Report the same for Control Limits.
- **Audits:** Report Internal and External Audits that were conducted. Include all relevant information such as which methods, by whom, corrective actions needed by when and discuss unresolved audit findings.
- **Performance Testing (PT) Samples:** Report the PT tests that are currently being tested with their due dates, report recent PT results by study, acceptable, total reported and the month and year.
- **Certifications:** Report on any certification programs being worked on by due date, packages completed. Describe any issues, lapses, or potential revocations.
- **Regulatory Updates:** Include information on new state or federal regulations that may impact the laboratory. Report new methods that require new instrumentation, deletion of methods, changes in sampling requirements and frequencies etc...

- **Miscellaneous:** Include any issues that may impact quality within the laboratory.
- **Next Month:** Report on plans for the upcoming month.
- **Lab Director Comments Section:** This section gives the Laboratory Director/Manager the opportunity to comment on issues discussed in the report and to document plans to resolve these issues. Unresolved issues that reappear in subsequent monthly reports must be commented on by the Laboratory Director/Manager.
- **Quality System Metrics Table:** The report also includes statistical results that are used to assess the effectiveness of the quality system. Effective quality systems are the responsibility of the entire laboratory staff. Each laboratory provides their results in a template provided by Corporate QA (Figure 17-2).

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The VP-QA/EHS 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 Analytical Division Senior Management Team and General Managers.

## **17.2 ANNUAL MANAGEMENT REVIEW**

The Senior Lab Management Team (Laboratory Director, Technical Director, Operations Manager, QA Manager, HR Supervisor, I.T. Supervisor) conducts an annual review 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. Corporate Operations and Corporate QA personnel may be included in this meeting at the discretion of the Laboratory Director/Manager. 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 cannot be solved by the lab and report them to Corporate IT.

This review uses information generated during the preceding year to assess the “big picture” by ensuring that routine quality actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review (refer to Section 17.1) 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 Management team 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.

The annual review includes the previous 12 months. Based on the annual review, 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)

The QA Manual is also reviewed at this time, and revised to reflect any significant changes made to the quality systems.

### **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. The Corporate Data Investigation/ Recall SOP shall be followed (SOP 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.

The Chairman/CEO, President/CEO, COOs, and Quality Directors receive a monthly report from the VP of Quality and EHS summarizing any current data integrity or data recall investigations as described in SOP CA-L-S-001. The General Managers are also made aware of progress on these issues for their specific labs.

## Figure 17-1.

### Example - QA Monthly Report to Management

LABORATORY: x  
PERIOD COVERED: Month/Year  
PREPARED BY: x      DATE: Month Day, Year  
DISTRIBUTED TO: xx (Include LD, GM, QA Director, etc...)

#### THREE KEY ISSUES FOR MONTH:

*Include a discussion of three key issues that were focused in on this month.*

1. x
  2. x
  3. x
- 

#### 1. METRICS

*Describe actions or improvement activities underway to address any outlying quality metrics.*

#### 2. SOPs

See Tab for SOP specifics.

The following SOPs were finalized (or reviewed for accuracy): (See Tab)

The following SOPs are due to QA: xx

In QA to complete: xx

#### 3. CORRECTIVE ACTION

Highlights: xx

Revised Reports:

*Describe the most frequent cause for report revisions and corrective/preventive action measures underway.*

Data Investigations/Recalls (Corporate Data Investigation/Recall SOP) :

*Include a discussion of any recalls handled at the lab level as Corp SOP.*

Client Feedback and Complaints:

*Include both positive and negative feedback.*

Describe the most serious client complaints) and resolutions in progress.

#### 4. MDLs AND CONTROL LIMITS

MDLs Due:

Control Limits Due:

**5. AUDITS**

INTERNAL AUDITS

*Discuss Any Outstanding Issues (or Attach Summary):*

EXTERNAL AUDITS

*Discuss Any Outstanding Issues (or Attach Summary):*

**6. PT SAMPLES**

The following PT samples are now in house (Due Dates):

xx

**7. CERTIFICATIONS**

Certification Packages Being Worked On (Include Due Date):

x

Describe any issues, lapses, or potential revocations.

**8. REGULATORY UPDATE**

*Include information on new state or federal regulations that may impact the laboratory – new methods that require new instrumentation, deletion of methods, changes in sampling requirements or frequencies, ...*

**9. MISCELLANEOUS**

*Include any issues that may impact quality within the laboratory.*

**10. NEXT MONTH**

*Items planned for next month.*

LAB DIRECTOR COMMENTS AND PLANNED CORRECTIVE ACTIONS:	
LAB DIRECTOR REVIEW:	DATE:

**Figure 17-2.**

**Example - Laboratory Metrics Categories**

No. of reports for month
No. of reports revised due to lab error
Percent revised reports
No. of Data Recall Investigations
No. of reports actually recalled
No. of Corrective Action Reports
No. of Corrective Action Reports still open
Total number of unresolved open Corrective Action Reports
Percent of unresolved open Corrective Action Reports
No. of reports independent QA reviewed
Percent QA Data Review: Reports
No. of technical staff (analysts/technicians, including temps)
No. of analyst work product reviewed year-to-date
No. of analytical instruments with electronic data file storage capability
No. of Analytical instruments reviewed for data authenticity year-to-date
Percent Analyst/Instrument Data Authenticity Audits
No. of client complaints
No. of client compliments
No. of planned internal audits
No. of planned internal method audits performed year-to-date
Percent annual Internal Audits complete
No. of open Internal Audit findings past due
Total Number of External Audit findings
No. of open External Audit findings past due
Percent External Audit findings past due
No. of PT analytes participated and received scores
No. of PT analytes not acceptable
Percent PT cumulative score
No. of PT repeat analyte failures cumulative (analyte failed more than once in 4 consecutive studies by PT type--only applies to failed analytes)
No. of SOPs

No. of SOPs reviewed/revised within 24 months
No. of methods or administrative procedures without approved SOPs
SOP status
Method certification losses due to performance/audit issues
Hold Time violations due to lab error
Date of last Comprehensive Ethics Training session
No. of staff that haven't received Comprehensive Ethics Training (>90 days from employment date)
MDL status (good, fair, or poor) >90%, >70%, <70%
Training Documentation Records (good, fair, or poor)
LQM Revision/review date
QAM updated to new integrated template
Last annual Internal Audit date (opened, closed)
Last Management QS Review date
No. of SOPs required for 12-month review cycle (DOD or drinking water)
No. of SOPs for 12-month cycle/revised within 12 months (Includes QS and Methods Listed in QSM)
12-month percent SOP status (Includes QS and Methods Listed in QSM)

## **SECTION 18**

### **PERSONNEL (NELAC 5.5.2)**

#### **18.1 OVERVIEW**

TestAmerica'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 Appendix 2.

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**

TestAmerica makes every effort to hire analytical staff 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. There are competent analysts and technicians in the industry who have not earned a college degree. 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 "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, colony counting, aseptic or quantitation techniques, etc. are also considered

As a general rule for analytical staff:

<b>Specialty</b>	<b>Education</b>	<b>Experience</b>
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 – <b><u>General</u></b>	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 – <b><u>Wet Chem</u></b> only (no advanced instrumentation)	Associate degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience

Specialty	Education	Experience
Technical Director - Microbiology	Bachelors degree in applied science with at least 16 semester hours in general microbiology and biology  An advanced (MS, PhD.) degree may substitute for one year of experience	And 2 years of 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 **TRAINING**

TestAmerica 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
New Hire Orientation	Immediately	All
Environmental Health & Safety Orientation	Day of hire	All
Environmental Health & Safety Orientation Follow-up Test	30-60 days after hire	All
Environmental Health & Safety Training	Refer to EH&S Manual	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 the employee's 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 CORP-QA-0013, Employee Orientation and Training.

#### **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 an Ethics Policy CA-L-P-001 and an Ethics Statement/Agreement (Appendix 1). All initial and annual training is documented by signature on the signed Ethics Statement/Agreement 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 (Appendix 1)
- 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--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 19

### ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS (NELAC 5.5.3)

#### 19.1 OVERVIEW

TestAmerica North Canton is an 11,466 sq ft 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 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. A 225KVA UPS is installed in the main electrical bus to provide at least 15 minutes of backup power in the event of a power failure. This unit also provides voltage and frequency control of lab and office power. A spike/surge arrestor is installed to protect against power surge/sag and lightning strikes. A 30 KW natural gas-fueled backup generator is installed to provide power to the I.T. area in the event of a power failure. Additionally, this generator provides power to two walk-in sample

storage coolers and several other smaller sample storage coolers. Smaller portable generators are available to provide “spot power” where needed in the event of a power failure.

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 (refer to Section 12).

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 3.

### **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 TestAmerica North Canton. 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.

Signs are posted in the laboratory designating employee only areas - "Authorized employees beyond this point".

## SECTION 20.0

### TEST METHODS AND METHOD VALIDATION (NELAC 5.5.4)

#### 20.1 OVERVIEW

TestAmerica North Canton 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)

TestAmerica North Canton 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 (refer to Section 6 on Document Control):

- All SOPs contain a revision number, effective date, and appropriate approval signatures. Controlled copies are available to all staff.
- Procedures for preparation, review, revision, and control are incorporated by reference to SOP CW-Q-S-002, Writing a Standard Operating Procedure (SOP), and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures.
- SOPs are reviewed at a minimum of every two 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. Refer to the corporate SOP CW-Q-S-002, Writing a Standard Operating Procedure, and SOP NC-QA-0027, Preparation and Management of Standard Operating Procedures, for content and requirements of technical and non-technical SOPs.

**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.

## **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, 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.

### **20.4.1 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.

In general, TestAmerica North Canton follows procedures from the referenced methods shown below in Section 20.3.1.4.

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.

20.4.1.1 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:

- Method 1664, Revision A: N-Hexane Extractable Material (HEM; Oil and Grease) and Silica Gel Treated N-Hexane Extractable Material (SGT-HEM); Non-polar Material) by Extraction and Gravimetry, EPA-821-R-98-002, February 1999
- Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water. Revised as of July 1, 1995. Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.

- *Standard Methods for the Examination of Water and Wastewater*, 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.
- *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.
- *Annual Book of ASTM Standards*, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- *Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261*

**TABLE 20-1. Wet Chemistry Methods <sup>1</sup>**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305. <sup>2</sup> SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water	305. <sup>2</sup> SM 2320 B		
	Solid	EPA 310.1 <sup>2</sup> (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Ash Content	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1 SM 5210 B	--	--
Bromide	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid	--	EPA 9081	--
Chemical Oxygen Demand	Water	EPA 410.4 SM 5220D	--	--
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 300.0A EPA 325.2 <sup>2</sup>	EPA 9056A EPA 9251	EPA 325.2 <sup>2</sup>
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	--
	Waste	EPA 3500-Cr-D	EPA 7196A	--
	Solid	--	EPA 3060A EPA 7196A	--
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5 <sup>2</sup> SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1 <sup>2</sup> SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1 <sup>2</sup> SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2 <sup>2</sup>	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M) <sup>2</sup> EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M) <sup>2</sup> EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2 <sup>2</sup>	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Waste	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Solid	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Water	SM 4500 NH <sub>3</sub> -E (Titration)	--	--

Analytical Parameters Matrix	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Nitrogen, Ammonia (cont'd)	Water	SM 4500 NH <sub>3</sub> -F (ISE)	--	--
Nitrite (NO <sub>2</sub> )	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO <sub>3</sub> )	Water	EPA 300.0A	EPA 9056A	SM 4500 NO <sub>3</sub> -E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO <sub>2</sub> /NO <sub>3</sub>	Water	EPA 353.2	--	--
	Waste	EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water	EPA 351.3	--	SM 4500 NO <sub>3</sub>
	Waste	EPA 351.3	--	--
	Solid	EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water	EPA 1664A	EPA 9071B	--
	Waste	EPA 1664A	EPA 9071B	--
	Solid	--	EPA 9071B	--
Ortho-phosphate o-PO <sub>4</sub>	Water	EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water	EPA 150.1 <sup>2</sup>	EPA 9040B	EPA 9041
	Waste	SM 4500 H-B	EPA 9045C	--
	Solid	---	EPA 9045C	--
Paint Filter	Water	--	EPA 9095A	--
Phenolics	Water	EPA 420.1	--	--
	Waste	--	EPA 9065	--
	Solid	--	EPA 9065	--
Phosphorus (Total)	Water	EPA 365.1	--	SM 4500 P-E
	Waste	EPA 365.1	--	--
	Solid	EPA 365.1	--	--
Sulfate (SO <sub>4</sub> )	Water	EPA 300.0A EPA 375.4 <sup>2</sup>	EPA 9056A EPA 9038	--
	Waste	EPA 300.0A (M) EPA 375.4 <sup>2</sup>	EPA 9056A EPA 9038	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1 <sup>2</sup>	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water	EPA 415.1 <sup>2</sup>	EPA 9060	SM 5310 D
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste	--	--	--
	Solid	--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	2540E
Total Suspended Solids	Water	EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	--
Turbidity	Water	EPA 180.1	--	--

<sup>1</sup> Any matrix not listed is not applicable for the associated method

<sup>2</sup> Removed from 40CFR

**TABLE 20-2. Methods for Mercury by Cold Vapor Atomic Absorption**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water	EPA 245.1	EPA 7470A	--
	TCLP Leachate	--	EPA 7470A	--
	Waste	--	EPA 7471A	--
	Solid	EPA 254.5	EPA 7471A	--

**TABLE 20-3. Methods for Mercury by Cold Vapor Atomic Fluorescence**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water	EPA 245.7	--	EPA 1631E

**TABLE 20-4. Methods for Metals by ICP and ICPMS**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Barium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---
Calcium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---
Cadmium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Iron	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water	EPA 200.7	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7	EPA 6010B EPA 6020	---
Manganese	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Selenium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Silver	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Tin	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Thallium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Vanadium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	---	EPA 6010B EPA 6020	---
Zinc	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

**TABLE 20-5. Metals Sample Preparation Methods**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	---	EPA 1311	---
	Waste	---	EPA 1311	---
	Solid	---	EPA 1311	---
ICP Metals	Water	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
ICPMS Metals	Water	EPA 200.8	EPA 3010A	---
	TCLP	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
CVAA Mercury	Water	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A	---
	Solid	---	EPA 7471A	---
CVAFS Mercury Low Level	Water	EPA 245.7	---	EPA 1631E

**TABLE 20-6. Organic Sample Preparation Methods**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	EPA 615	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water	---	EPA 5030B	WI GRO
	Waste	---	EPA 5030B EPA 5035	WI GRO
	Solid	---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water	---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	WI DRO
	Solid	---	EPA 3550B	WI DRO

**TABLE 20-7. Organic Methods of Analysis**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 8260B	---
	Waste	---	EPA 8260B	---
	Solid	---	EPA 8260B	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 8270C	---
	Waste	---	EPA 8270C	---
	Solid	---	EPA 8270C	---
Pesticides/PCBs by GC	Water	EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate	---	Pesticides 8081A PCBs 8082	---
	Waste	---	Pesticides 8081A PCBs 8082	---
	Solid	---	Pesticides 8081A PCBs 8082	---
Phenoxyacid Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Gasoline Range Organics by GC	Water	---	EPA 8015B (M)	WI GRO
	Waste	---	EPA 8015B (M)	---
	Solid	---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water	---	EPA 8015B (M)	WI DRO
	Waste	---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water	---	---	SOP
Formaldehyde Carbonyl Compounds	Water	---	EPA 8315	---

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 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.

20.4.2.1 A demonstration of capability is performed whenever there is a change in instrument type, method or personnel.

20.4.2.2 The initial demonstration of capability must be thoroughly documented and approved by the Technical Director and QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratories archiving procedures. Refer to Section 15, Control of Records.

20.4.2.3 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).
- The reporting limit is set at or above the first standard of the curve for the analyte.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds. This must be addressed in the Case Narrative.

- Refer to Section 12, Control of Non-Conforming Work.

### **20.4.3 Initial Demonstration of Capability (IDOC) Procedures**

- 20.4.3.1 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).
- 20.4.3.2 Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest. Refer to SOP CORP-QA-0013, Employee Orientation and Training, for details on this procedure.

A certification statement (see Figure 20-1 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.

## **20.5 LABORATORY DEVELOPED METHODS AND NON-STANDARD METHODS**

Any new method developed by the laboratory must be fully defined in an SOP/Methods Manual (Section 20.2) 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. The information included in the checklist below (Figure 20-2) is needed before samples are accepted for analysis by a new 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 (from 2003 NELAC Standard).

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. The laboratory determinations of MDLs are described in Section 20.6.

#### 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 level at which both the presence of an analyte and its concentration can be reliably determined. 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, a determination of the applicable range of the method may be performed. In most cases, range is determined and demonstrated by comparison of the response of an analyte in a curve to established or targeted criteria. The curve is used to establish the range of quantitation and the lower and upper values of the curve represent upper and lower quantitation limits. Curves are not limited to linear relationships.

#### 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 Section 20.7.10). 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.

**20.7.1** MDLs are initially performed for each individual instrument and non-microbiological method analysis. Unless there are requirements to the contrary, the laboratory will use the highest calculated MDL for all instruments used for a given method as the MDL for reporting purposes. This MDL is not required for methods that are not readily spiked, e.g., pH, turbidity, etc. Titration and gravimetric methods where there is no additional preparation involved, the MDL is based on the lowest discernable unit of measure that can be observed.

**20.7.2** MDLs must be run against acceptable instrument QC, including ICVs and Tunes. This is to ensure that the instrument is in proper working condition and falsely high or low MDLs are not calculated.

**20.7.3** Use only clean matrix which is free of target analytes (e.g.: Laboratory reagent water, Ottawa Sand) unless a project specific MDL is required in a field sample matrix.

**20.7.4** The Reporting Limit should generally be between two and five times the MDL. If the MDL is being performed during method development, use this guideline to determine the Reporting Limit for the analysis. For DoD labs, the RL is  $\geq 3x$  MDL. If a sample is diluted, the reported MDL is adjusted according to the dilution factor.

**20.7.5** If the MDL is < 1/10 of the spike concentration for more than 10% of the analytes in the method (< 1/5 of spike recovered for DoD for water samples), the MDL must be repeated (including extraction or digestion) using a lower spike level unless the percent recovery is <50% or >150% of the “true value”. Note: The concentration of the spike will be at a level below the calibration range. Note: The spiking concentration must be less than the reporting limit.

**20.7.6** The calculated MDL cannot be not greater than the spike amount.

**20.7.7** If the most recent calculated MDL does not permit qualitative identification of the analyte then the laboratory may use technical judgment for establishing the MDL (e.g., calculate what level would give a qualitative ID, compare with IDL (20.7), spike at a level where qualitative ID is determined and assign that value as MDL, minimum sensitivity requirements, Standard deviation of method blanks over time, etc.).

**20.7.8** Each of the seven spikes must be qualitatively identifiable, e.g., appear in both columns for dual column methods, characteristic ions for GCMS mass spectra, etc. Manual integrations to force the baseline for detection are not allowed.

**20.7.9** The initial MDL is calculated as follows:

$$\text{MDL} = t_{(n-1, 1-a = 0.99)} \times (\text{Standard Deviation of replicates})$$

where  $t_{(n-1, 1-a = 0.99)} = 3.143$  for seven replicates.

**20.7.10** Subsequent to the initial MDL determination, periodic MDL verification, confirmation or determinations may be performed by the procedure in 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices (e.g., method blanks over time, single standard spikes that have been subjected to applicable sample prep processes, etc.). The procedures utilized must be documented in the MDL SOP NC-QA-0021, Evaluation of Method Detection Limits for Chemical Tests.

**20.7.11** Because of the inherent variability in results outside of the calibration range, TestAmerica does not recommend the reporting of results below the lowest calibration point in a curve; however, it is recognized that some projects and agencies require the reporting of results below the RL. Any result that falls between the MDL and the Reporting limit, when reported, will be qualified as an estimated value.

**20.7.12** Detections reported down to the MDL must be qualitatively identified.

**20.7.13** MDLs and Reporting limits are adjusted in LIMs based on moisture content and sample aliquot size.

## **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 by using seven replicate spike analyses, like MDL but without sample preparation, or by the analysis of ten instrument blanks and calculating three times the absolute value of the standard deviation.

**20.8.3** If IDL is > than the MDL, it may be used as the reported MDL.

## **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 two times the calculated MDL. The analytes must be qualitatively identified or see Section 20.6.7 for other options. This verification does not apply to methods that are not readily spiked, e.g., pH, turbidity, etc. 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 (see Section 20.6.7). MDLs must be verified at least quarterly.

**20.9.2** When a Reporting limit is established, it must be initially verified by the analysis of a low-level standard or QC sample (LCS at 1-2 the reporting limit) and annually thereafter. Unless there are requirements to the contrary, the acceptance criteria is  $\pm 50\%$ . The annual requirement is waived for methods that have an annually verified MDL.

## **20.10 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 in each department.

For GC, HPLC and IC methods, there must be sufficient separation between analyte peaks so as to not misidentify analytes. In the mid-level standard, the distance between the valley and peak height cannot be any less than 25% of the sum of the peak heights of the analytes. This also applies to GCMS in the case where the two compounds share the same quantitation ion.

**Note:** Some analytes do not separate sufficiently to be able to identify or quantitate them as separate analytes, e.g., m-xylene and p-xylene, and are quantitated and reported as a single analyte, e.g., m,p-xylenes.

Once the analyst has determined that the instrument is in optimum working condition through calibration and calibration verification procedures, he or she uses a mid-range calibration or calibration verification standard to establish the retention times for each of the individual analytes in a method. The analyst makes three injections of the same standard over a 72-hour (24-hr period for 300.0) period, tabulating the retention times for each analyte for each of the three injections. The width of retention time window is normally the average absolute retention time  $\pm 3$

Standard Deviations. A peak outside the retention time window will not be identified by the computer as a positive match of the analyte of interest.

It is possible for the statistically calculated RT window to be too tight and need to be adjusted based on analyst experience. In these instances method default retention time windows may be used, e.g., for 8000 series methods a default of 0.03 minutes may be used, and EPA CLP 0.05 minutes is used. The same concept is applied when any peak outside of that window will not be identified by the computer as a positive match.

The calibration verification standard at the beginning of a run may be used to adjust the RT for an analyte. This is essentially re-centering the window, but the size of the window remains the same. The RTs are verified when all analytes are within their RT windows and are properly identified.

## **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, atomic absorption, or fluorescence profiles.

## **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  $k=2$ .

**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 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). 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 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.

#### **20.13.1 Computer and Electronic Data Related Requirements**

The three basic objectives of our computer security procedures and policies are shown below. The laboratory is currently running the QuantIMS LIMS 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 AS400 which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

20.13.1.1 **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.

**Note:** "Commercial off-the-shelf software in use within the designed application range is considered to be sufficiently validated" from *NELAC 2003 Standard*. However, laboratory specific configurations or modifications are validated prior to use.

- In order to assure accuracy, all data entered or transferred into the LIMS data system goes through a minimum of two levels of review.
- The QA department performs random data audits to ensure the correct information has been reported.

- Changes to reports are documented via Change Order Forms and by noting "Revised" on the cover page.
- Analytical data file security is provided through three policies.
  - The first policy forbids unauthorized personnel from using laboratory data acquisition computers.
  - The second policy is the implementation of network passwords and login names that restrict directory access.
  - The third layer is maintained through the LIMS and includes the use of username/password combinations to gain access to the LIMS system, the fact that all data in the LIMS is associated with the user to added/reviewed the data, and the restriction of review authority of data.
- All software installations will be in accordance with any relevant copyright licensing regulations.
- All software installed on any computer within the laboratory must be approved by the Information Technology Department regional support technician assigned to the laboratory. Shrink-wrapped or otherwise sealed OEM software that is directly related to instrument usage does not need approval, but the Information Technology Department must be notified of the installation.
- Anti-virus software shall be installed on all servers and workstations. The anti-virus software shall be configured to check for virus signature file and program updates on a daily basis and these updates will be pushed to all servers and workstations. The anti-virus software will be configured to clean any virus-infected file if possible, otherwise the file will be deleted. Disks and CDs brought from any outside source that are not OEM software must be scanned for viruses before being accessed.
- **Interlab LIMS Permissions Policy**
  - PURPOSE - The purpose of this policy is to provide a mechanism for maintaining the integrity of information contained in each laboratory's LIMS while providing the necessary access for information sharing to staff at other laboratory facilities.
  - DEFINITIONS - Host Laboratory: The laboratory facility that 'owns' the LIMS system or 'hosts' a project/job.
  - POLICIES
    - (a) All permissions for the laboratory's LIMS system must only be granted by a representative of that laboratory.
      - If someone outside of the host lab needs permissions for Project Management or other uses, they must go through the Lab Director or his/her designated representative.
      - Permissions must never be granted without the knowledge of the host laboratory.
    - (b) Only laboratory analytical or QA staff from the home laboratory may have edit permissions for laboratory analysis data.

(c) Any changes made in laboratory's LIMS system:

- Must be documented and traceable.
- If made by staff of an affiliate lab, written permission from the home lab to make the changes (email approval is sufficient) is required.
- No corrections may be made in another laboratories system without their knowledge.

(d) Data qualifiers in laboratory reports must only be corrected, edited, etc. by the staff at the host laboratory.

(e) Full analytical data "View" only permissions may be granted to outside Project Management and Sales staff. Search permissions may also be granted so status may be checked.

(f) All qualifiers must be approved by QA staff before adding to standard reference tables.

**(g) Please contact Corporate QA or IT staff if you have any questions regarding implementation or interpretation of this policy.**

20.13.1.2 **Ensure Information Availability:** Protection against loss of information or service through scheduled back-ups, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

- Insured by timely backup procedures on reliable backup media, stable file server network architecture, and UPS protection
- UPS Protection:
  - Each fileserver is protected by an appropriate power protection/backup unit. In the event of a power outage, there is approximately 15-30 minutes of up-time for the servers prior to shutdown. This allows for proper shutdown procedures to be followed with the file servers.
- File Server Architecture
  - All files are maintained on multiple Windows 2000 or newer servers which are secured physically in the Information Technology office. Access to these servers is limited to members of the Information Technology staff.
  - All supporting software is maintained for at least five years from the last raw data generated using that software. Length of time is dependent on local regulations or client requirements, e.g., OVAP requires ten years.
- System Back-up Overview and Procedures
  - Data from both servers and instrument attached PC's are backed up and purged in compliance with the corporate back-up policy.
  - A Maintenance Plan has been defined to create a daily archive of all data within the LIMS database to a backup location. This backup is initiated automatically by either the database or back-up system.

- Backup tapes will be stored in compliance with the corporate Data Backup Policy. Backup verifications are carried out in accordance with the corporate Data Backup Policy.
- Instrument data back-ups are verified on a periodic basis by the QA department when performing electronic data audits. The audit takes place on data that has been moved to a back-up location ensuring that it has been moved.

20.13.1.3 Maintain Confidentiality: Ensure data confidentiality through physical access controls, and encryption of when electronically transmitting data.

- All servers are located in a secure area of the IT department offices. Access to the servers is limited to IT staff members.
- The company website contains SSL (Secure Socket Layer) encryption for secure website sessions and data transfers.
- Electronic documents such as PDF files and electronic data deliverables may be made available to clients via the secure web site. The logon page for this web site contains an agreement that the customer must accept before they will be logged on which states that the customer agrees not to alter any electronic data made available to them.

### 20.13.2 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 peer review once updated in LIMS. The review checklists are signed by both the analyst and 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 must 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.

20.13.2.1 All raw data must be retained. 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/year). It must be easily identifiable who performed which tasks if multiple people were involved.

20.13.2.2 In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ( $\mu\text{g/l}$ ) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ( $\mu\text{g/kg}$ ) for solids. The units “mg/l” and “mg/kg” are the same as “parts per million (ppm)”. The units “ $\mu\text{g/l}$ ” and “ $\mu\text{g/kg}$ ” are the same as “parts per billion (ppb).” For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%.

- Several environmental methods, such as color, turbidity, conductivity, use very specific, non-concentration units to report results (e.g., NTU, umhos/cm etc).
- Occasionally, the client requests that results be reported in units which take into account the measured flow of water or air during the collection of the sample. When they provide this information, the calculations can be performed and reported.

20.13.2.3 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.

20.13.2.4 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.

### **20.13.3 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 QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

#### **20.13.4 Review / Verification Procedures**

20.13.4.1 Data Recording Procedures: To ensure data integrity, all documentation of data and records generated or used during the process of data generation must be performed in compliance with Section 3 of this document and Policy T-Q-005, Recording Laboratory Observations and Raw Data.

20.13.4.2 Data Reduction and Verification Procedures: Data review procedures comprise a set of computerized and manual checks applied at appropriate levels of the measurement process. Data review begins with the reduction or processing of data and continues through verification of the data and the reporting of analytical results. Calculations are checked from the raw data to the final value prior to reporting results for each group of samples. Data reduction can be performed by the analyst who obtained the data or by another analyst. Data verification starts with the analyst who performs a 100% review of the data to ensure the work was done correctly the first time. Data verification continues with review by a second reviewer who verifies that data reduction has been correctly performed and that the analytical results correspond to the data acquired and processed.

20.13.4.2.1 Data Reduction and Initial Verification: Data reduction and initial verification may be performed by more than one analyst depending upon the analytical method employed. The preparation and analytical data may be reviewed independently by different analysts. In these instances, each item may not be applicable to the subset of the data verified or an item may be applicable in both instances. It is the responsibility of the analyst to ensure that the verification of data in his or her area is complete. The data reduction and initial verification process must ensure that:

- Sample preparation information is correct and complete including documentation of standard identification, solvent lot numbers, sample amounts, etc.
- Analysis information is correct and complete including proper identification of analysis output (charts, chromatograms, mass spectra, etc.)
- Analytical results are correct and complete including calculation or verification of instrument calibration, QC results, and qualitative and quantitative sample results with appropriate qualifiers
- The appropriate SOPs have been followed and are identified in the project and/or laboratory records
- Proper documentation procedures have been followed
- All non-conformances have been documented
- Special sample preparation and analytical requirements have been met.
- The data generated have been reported with the appropriate number of significant figures as defined by the analytical method in the LIMS or otherwise specified by the client.

In general, data will be processed by an analyst in one of the following ways:

- Manual computation of results directly on the data sheet or on calculation pages attached to the data sheets
- Input of raw data for computer processing

- Direct acquisition and processing of raw data by a computer.

If data are manually processed by an analyst, all steps in the computation shall be provided including equations used and the source of input parameters such as response factors (RFs), dilution factors, and calibration constants. If calculations are not performed directly on the data sheet, they may be attached to the data sheets.

Manual integrations are sometimes necessary to correct misintegrations by an automatic data system software program, but must only be performed when necessary. Further discussion of manual integrations and the required documentation is given in Policy S-Q-004, Acceptable Manual Integration Practices.

For data that are input by an analyst and processed using a computer, a copy of the input shall be kept and uniquely identified with the project number and other information as needed. The samples analyzed must be clearly identified.

If data are directly acquired from instrumentation or a test procedure and processed, or immediately entered into LIMS, the analyst must verify that the following are correct:

- Project and sample numbers
- Calibration constants and RFs
- Units
- Numerical values used for reporting limits.

Analysis-specific calculations for methods are provided in SOPs. In cases where computers perform the calculations, software must be validated or verified, as described in Section 6.0 of this document, before it is used to process data.

The data reduction is documented, signed and dated by the analyst completing the process. Initial verification of the data reduction by the same analyst is documented on a data review checklist, signed and dated by the analyst.

20.13.4.2.2. Data Verification: Following the completion of the initial verification by the analyst performing the data reduction, a systematic check of the data that has been fully reduced and checked through Level 1 review is performed by an experienced peer, supervisor, or designee. This Level 2 check is performed to ensure that Level 1 review has been completed correctly and thoroughly. The second level reviewer examines the data signed by the analyst. Any exceptions noted by the analyst must be reviewed. Included in this review is an assessment of the acceptability of the data with respect to:

- Adherence of the procedure used to the requested analytical method SOP
- Correct interpretation of chromatograms, mass spectra, etc.
- Correctness of numerical input when computer programs are used (checked randomly)
- Correct identification and quantitation of constituents with appropriate qualifiers

- Numerical correctness of calculations and formulas (checked randomly)
- Acceptability of QC data (100% review)
- Documentation that instruments were operating according to method specifications (calibrations, performance checks, etc.)
- Documentation of dilution factors, standard concentrations, etc.
- Sample holding time assessment.

This review also serves as verification that the process the analyst has followed is correct in regard to the following:

- The analytical procedure follows the methods and client-specific instructions.
- Nonconforming events have been addressed by corrective action as defined on a nonconformance memo
- Valid interpretations have been made during the examination of the data and the review comments of the initial reviewer are correct
- The package contains all of the necessary documentation for data review and report production and results are reported in a manner consistent with the method used for preparation of data reports.

The specific items covered in the second stage of data verification may vary according to the analytical method, but this review of the data must be documented by signing the same checklist.

20.13.4.2.3. Completeness Verification: A third-level review is performed by the reporting and project management staff. This review is required before results are submitted to clients. This review serves to verify the completeness of the data report and to ensure that project requirements are met for the analyses performed. The items to be reviewed are:

- Analysis results are present for every sample in the analytical batch, reporting group, or sample delivery group (SDG)
- Every parameter or target compound requested is reported with either a value or reporting limit
- All nonconformances, including holding time violations, and data evaluation statements that impact the data quality are accompanied by clearly expressed comments from the laboratory
- The final report contains all the supporting documentation required by the project, and is in either the standard TestAmerica format or in the client-required format.
- Implement checks to monitor the quality of laboratory results using correlation of results for different parameters of a sample (for example, does the TOC results justify the concentration of organic compounds found by GC/MS.)
- A narrative to accompany the final report will be finalized by the PM. This narrative will include relevant comments collected during the earlier reviews.

#### 20.13.5 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 SOP CA-Q-S-002 as the guidelines.

- 20.13.5.1 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.
- 20.13.5.2 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.
- 20.13.5.3 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.
- 20.13.5.4 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 20-1.**  
**Example - Demonstration of Capability Documentation**

## ***Analyst Demonstration of Capability*** **Certification Statement**

**Analyst Name**  
**Date:**

*Test Method:*  
*SOP:*  
*Matrix:*

TestAmerica North Canton laboratory  
4101 Shuffel Drive NW  
North Canton, OH 44720  
(330) 497-9396

We, the undersigned, CERTIFY that:

1. The analyst identified above, using the cited test method with the specifications in the cited SOP, which is in use at this facility for the analysis of samples under the TestAmerica Quality Assurance Plan, has met the Initial or Ongoing Demonstration of Capability.
2. The test method was performed by the analyst identified on this certification following the TestAmerica SOP.
3. A copy of the laboratory-specific SOP is available for all personnel on-site.
4. The data associated with the initial/ongoing demonstration of capability are true, accurate, complete and self-explanatory (\*). These data are attached to this certification statement.
5. 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 well organized and available for review by authorized inspectors.

Comments/Observations:

Analyst's Name	Signature	Date
----------------	-----------	------

Technical Director's Name	Signature	Date
---------------------------	-----------	------

QA Manager's Name	Signature	Date
-------------------	-----------	------

\* *True: Consistent with supporting data.*

*Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.*

*Complete: Includes the results of all supporting performance testing.*

*Self-explanatory: Data properly labeled and stored so that the results are traceable and require no additional explanation.*

**Figure 20-2.**

**Example - New Method / Additional Analyte Checklist**

**New Method / Additional Analyte Checklist**

The following items are **required** to be completed prior to the acceptance of client samples.

Fill in any blanks that do not apply with "NA".

Forward to Analytical Group, Operations Manager, Technical Director, and QA.

New Method – include method number and reference \_\_\_\_\_

New Analytes – list analyte and reporting limit

---



---

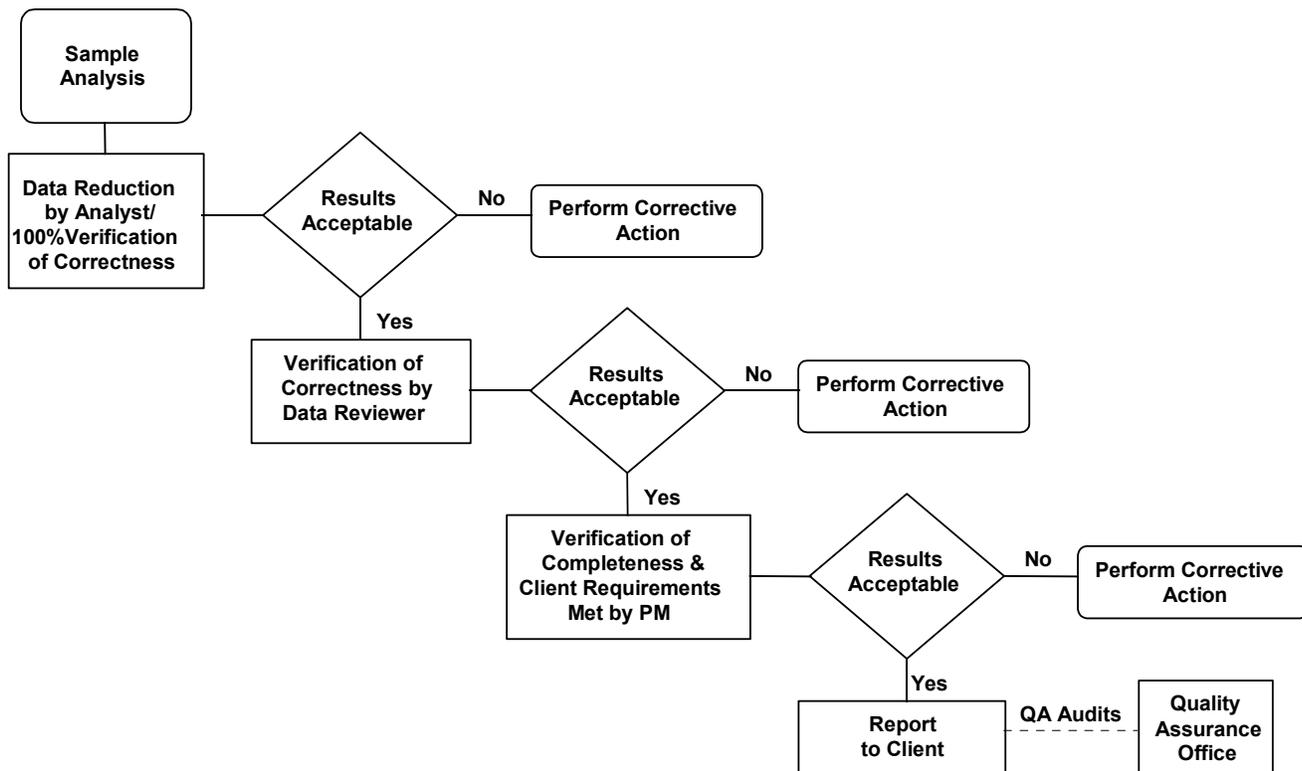


---

Matrix: water – solid – waste	
Analytical Groups Contacted	
Standard available for new analyte(s), including second source. If not, include time frame for order.	
Cost	
MDL study required	
MDL study completed	
LCS/MS/MSD spike required	
Project tied to QAPP? Lab has a copy?	
Project Program: DoD QSM, LCG, Client specific, State Specific	
Special QC Requirements: control limits, special criteria	
Certification required	
Standard Operating Procedure available for method	
One time project or on-going	
Special reporting parameters	
SAC created in LIMS	
Spike lists created in LIMS	
RL/MDL data entered into LIMS	

**QA Review / Acceptance** \_\_\_\_\_ **Date** \_\_\_\_\_

**Figure 20-3.**  
**Work Flow**



## SECTION 21

### EQUIPMENT (AND CALIBRATIONS) (NELAC 5.5.5)

#### 21.1 OVERVIEW

TestAmerica 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 21-1.

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** TestAmerica North Canton follows a well-defined 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 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.

21.2.2.1 Calibrations, routine maintenance, and adjustments are part of the Analyst and Group Leader responsibilities. However, service contracts may be in place for some instruments to cover any major repairs.

21.2.2.2 High purity gases, reagents, and spare parts are kept on hand to minimize repair time and optimize instrument performance.

**21.2.3** Table 21-2 summarizes the schedule for routine maintenance. It is the responsibility of each Group Leader 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 Logbooks 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, 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.)

21.2.4.3 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 it is clear that a page is missing if only half a signature is found in the logbook.

**21.2.5** In addition, the maintenance records contain:

- The identification of the instrument/equipment (instrument Serial and Model Number)
- The date the instrument/equipment was put into use.
- If available, the condition when the instrument was received, e.g. new, used, reconditioned

**21.2.6** 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 (refer to Sections 12 and 13).

**21.2.7** 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 using the procedures outlined in Section 8.

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      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, field sampling devices, temperature measuring devices, 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.

Each balance is checked prior to 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 five 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. Reference SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration. A list of balances is in Table 21.2.

#### **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 Thermometers**

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 three 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 has increments of 0.2 °C, and has a range applicable to all 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 logsheets. Monitoring method-specific temperatures, including incubators, heating blocks, water baths, and ovens, is documented in method-specific logsheets. More information on this subject can be found in SOP NC-QA-015, Equipment Monitoring and Thermometer Calibration.

### **21.3.4 Refrigerators/Freezer Units, Waterbaths, Ovens and Incubators**

The temperatures of all refrigerator units and freezers used for sample and standard storage are monitored each working day (seven days a week for DOD labs).

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 or  $4 \pm 2^{\circ}\text{C}$ .

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 Logsheets posted on each unit and method-specific logbooks.

### **21.3.5 Autopipettors, Dilutors, and Syringes**

Mechanical volumetric dispensing devices including burettes (except Class A Glassware) are checked for accuracy at least quarterly. Glass micro-syringes are considered the same as Class A glassware.

The laboratory maintains a sufficient inventory of autopipettors, and dilutors of differing capacities that fulfill all method requirements.

These devices are given unique identification numbers, and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

Any device not regularly verified cannot 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.

### **21.3.6 Field Sampling Devices (ISCO Autosamplers)**

Each autosampler (ISCO) is assigned a unique identification number in order to keep track of the calibration. This number is recorded on the sampling documentation in a logbook.

The autosampler is calibrated semi-annually by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The autosampler is programmed to run three cycles, and each of the three cycles is measured into a beaker to verify 100 ml are received.

If the RSD (Relative Standard Deviation) between the three cycles is greater than 20%, the procedure is repeated. If the result is still greater than 20%, the following options may be employed:

- 1) The unit is taken out of service.
- 2) The unit is used to pull composite samples only over a 24-hour period.

The results of this check are kept in a logbook/binder.

## **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.

## **CALIBRATION STANDARDS**

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. However, the general procedures are described below.

- 21.4.1.1 For each analyte and surrogate (if applicable) of interest, prepare calibration standards at the minimum number of concentrations as stated in the analytical methods. If a reference or mandated method does not specify the number of calibration standards, the minimum number is three, not including blanks or a zero standard. All of the standard solutions are prepared using Class A volumetric glassware, calibrated pipettes, and/or microsyringes and appropriate laboratory quality solvents and stock standards.
- 21.4.1.2 Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to NIST whenever possible. Dilution standards are prepared from stock standards purchased from commercial suppliers. A standard log is maintained for each department, containing concentration, date of receipt, date of standard preparation, any dilutions made, lot number, supplier, type of solvent and a unique code number to identify the standard.
- 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 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 lowest calibration standard must be at or below the reporting limit. The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.
- 21.4.1.5 Given the number of target compounds addressed by some of the organic methods, it may be necessary to prepare several sets of calibration standards, each set consisting of the appropriate number of solutions at different concentrations. The initial calibration will then involve the analysis of each of these sets of the appropriate number of standards.
- 21.4.1.6 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 FOR ORGANIC METHODS (GC, HPLC, GC/MS)**

21.4.2.1 Many of the organic analytical methods utilize an internal standard calibration (GCMS and some GC). Because of the complex nature of the multiplex chromatograms produced by the method, some instruments necessitate the use of external standard calibration (most GC and HPLC). Surrogate compounds are included in the calibration processes for all appropriate organic analyses. For more details on the calibration types listed below, refer to SOP CA-Q-S-005, Calibration Curves.

21.4.2.2 Once the operating parameters have been established according to the method, each instrument is calibrated for the appropriate method. The analyst prepares five or more standard solutions at various concentrations containing all of the analytes of interest, internal standards, and surrogates that are appropriate for the method. Note: There are a several EPA methods that have different requirements and are exceptions (e.g. EPA 547) where a minimum of three calibration standards are prepared and analyzed.

21.4.2.3 The standard solutions are introduced into the instrument in the same manner as samples are; whether it be by direct injection, by headspace analysis, or by purge and trap. The calibration factor (CF) for methods that use external standards, and the response factor (RF) for methods that use internal standards are calculated for the five standards.

- External standard calibration involves comparison of instrument responses from the sample to the responses from the target compounds in the calibration standards. Sample peak areas (or peak heights) are compared to peak areas (or heights) of the standards. The ratio of the response to the amount of analyte in the calibration standard is defined as the Calibration factor (CF).
- Internal standard calibration involves the comparison of instrument responses from the target compounds in the sample to the responses of specific standards added to the sample or sample extract prior to injection. The ratio of the peak area (or height) of the target compound in the sample or sample extract to the peak area (or height) of the internal standard in the sample or sample extract is compared to a similar ratio derived for each calibration standard. The ratio is termed the response factor (RF), and may also be known as a relative response factor in other methods.

In many cases, internal standards are recommended. These recommended internal standards are often brominated, fluorinated, or stable isotopically labeled analogs of specific target compounds, or are closely related compounds whose presence in environmental samples is highly unlikely. The use of specific internal standards is available in the method SOP.

Whichever internal standards are employed, the analyst needs to demonstrate that the measurement of the internal standard is not affected by method analytes and surrogates or by matrix interferences. In general, internal standard calibration is not as useful for GC and HPLC methods with non-MS detectors because of the inability to chromatographically resolve many internal standards from the target compounds. The use of MS detectors makes internal standard calibration practical because the masses of the internal standards can be resolved from those of the target compounds even when chromatographic resolution cannot be achieved.

When preparing calibration standards for use with internal standard calibration, add the same amount of the internal standard solution to each calibration standard, such that the concentration of each internal standard is constant across all of the calibration standards, whereas the concentrations of the target analytes will vary. The internal standard solution will contain one or more internal standards and the concentration of the individual internal standards may differ within the spiking solution. Not all internal standards need to be at the same concentration in this solution. The mass of each internal standard added to each sample extract immediately prior to injection into the instrument or to each sample prior to purging must be the same as the mass of the internal standard in each calibration standard. The volume of the solution spiked into sample extracts should be such that minimal dilution of the extract occurs, e.g., 10 uL of solution added to a 1 mL final extract results in only a negligible 1% change in the final extract volume which can be ignored in the calculations.

An ideal internal standard concentration would yield a response factor of one for each analyte. However, this is not practical when dealing with more than a few target analytes. Therefore, as a general rule, the amount of internal standard should produce an instrument response, e.g., area counts, that is no more than 100 times that produced by the lowest concentration of the least responsive target analyte associated with the internal standard. This should result in a minimum response factor of approximately 0.01 for the least responsive target compound. Refer to SOP CA-Q-S-005, Calibration Curves, for specific calculations.

21.4.2.4 Policies regarding the use of calibration standard results for creating the calibration curve are as follows:

- A low calibration standard may be excluded from the calibration if the signal-to-noise ratio or spectral criteria are not suitable. The reporting level must be elevated to be the lowest calibration standard used for calibration.
- The upper calibration standard may be excluded if it saturates the detector or is obviously becoming non-linear. Any sample exceeding the upper standard used in the calibration must be diluted and re-analyzed.
- Mid-calibration standards may not be excluded unless an obvious reason is found, i.e., cracked vial, incorrectly made, etc. The failed standard should be re-run immediately and inserted into the initial calibration. If not useful, recalibration is required.

#### 21.4.2.5 **Percent RSD Corrective Action**

Given the potentially large numbers of analytes that may be analyzed in some methods, it is likely that some analytes may exceed the acceptance limit for the RSD for a given calibration. In those instances, the following steps are recommended, but not required.

- 21.4.2.5.1** The first step is generally to check the instrument operating conditions. This option will apply in those instances where a linear instrument response is expected. It may involve some trade-offs to optimize performance across all target analytes. For instance, changes to the operating conditions necessary to achieve linearity for problem compounds may cause the RSD for other

compounds to increase, but as long as all analytes meet the RSD limits for linearity, the calibration is acceptable.

**21.4.2.5.2** If the RSD for any analyte is greater than the applicable acceptance criteria in the applicable analytical method, the analyst may wish to review the results (area counts, calibration or response factors, and RSD) for those analytes to ensure that the problem is not associated with just one of the initial calibration standards. If the problem appears to be associated with a single standard, that one standard may be reanalyzed and the RSD recalculated. Replacing the standard may be necessary in some cases.

**21.4.2.5.3** A third alternative is to narrow the calibration range by replacing one or more of the calibration standards with standards that cover a narrower range. If linearity can be achieved using a narrower calibration range, document the calibration linearity, and proceed with analyses. The changes to the upper end of the calibration range will affect the need to dilute samples above the range, while changes to the lower end will affect the overall sensitivity of the method. Consider the regulatory limits or action levels associated with the target analytes when adjusting the lower end of the range.

**Note:** When the purpose of the analysis is to demonstrate compliance with a specific regulatory limit or action level, the laboratory must ensure that the method quantitation limit is at least as low as the regulatory limit or action level.

21.4.2.6 Alternatively, the least squares regression may be used to determine linearity. A five-point line must result in a correlation coefficient ( $r$ ) of 0.990 or better using the least squares method to be considered acceptable.

21.4.2.7 Instead of a linear curve model (either Average RF or least squares regression), a second order curve (Quadratic) may be used (and preferred) as long as it contains at least six data points. As a rule of thumb, if there is a consistent trend in RFs (or CFs) in the calibration curve, either up or down, then quadratic curve fit may be indicated as the preferred calibration routine for that analyte. The coefficient of determination (COD or  $r^2$ ) for the quadratic curve must be at least 0.99 for it to be considered acceptable. For more details on the calculations see Calibration Curve SOP CA-Q-S-005. Some limitations on the use of quadratic curve fits:

**21.4.2.7.1** Care **MUST** be exercised to assure that the results from this equation are real, positive, and fit the range of the initial calibration.

**21.4.2.7.2** They **may not** be used to mask instrument problems that can be corrected by maintenance. (Not to be used where the analyte is normally found to be linear in a properly maintained instrument).

**21.4.2.7.3** They **may not** be used to compensate for detector saturation. If it is suspected that the detector is being saturated at the high end of the curve, remove the higher concentration standards from the curve and try a 1<sup>st</sup> order fit or average RF.

### **21.4.3 Calibration for Inorganic Analyses**

EPA Method 7000 from EPA SW-846 is a general introduction to the quality control requirements for metals analysis. For inorganic methods, quality control measures set out in the individual methods and in the *Standard Methods for the Examination of Water and Wastewater* (20th Edition) may also be included. Standard Operating Procedures for the analysis and the quality control documentation measures are available electronically on the public drive.

In general, inorganic instrumentation is calibrated with external standards. Some exceptions would be Inductively Coupled Plasma (ICP), Inductively Coupled Plasma Mass Spec (ICPMS), and Ion Chromatography Mass Spec (ICMS). These analyses may use an internal standard to compensate for viscosity or other matrix effects. While the calibration procedures are much the same for inorganics as they are for organics, CF's or RF's are not used. The calibration model in Section 21.4.2.6 is generally used for most methods, however in some instances the model from Section 21.4.2.7 may be used. A correlation coefficient ( $r$ ) of 0.995 or greater must be used to accept a calibration curve generated for an inorganic procedure. Correlation coefficients are determined by hand-held scientific calculators or by computer programs [state what your lab uses] and documented as part of the calibration raw data. Coefficients of calibration curves used for quantitation must be documented as part of the raw data. Curves are not allowed to be stored in calculator memories and must be written on the raw data for the purposes of data validation.

21.4.3.1 "Calibrations" for titrimetric analyses are performed by standardizing the titrants against a primary standard solution. See specific methods in *Standard Methods for the Examination of Water and Wastewater* (20th Edition) for more information.

21.4.3.2 Spreadsheets that are used for general chemistry calculations must have all cells containing calculations locked to prevent accidental changes to the calculations.

21.4.3.3 Instrument technologies, e.g., ICP, with validated techniques from the instrument manufacturer or other methods using a zero point and single point calibration require the following:

**21.4.3.3.1** The instrument is calibrated using a zero point and a single point calibration standard.

**21.4.3.3.2** Sample results within the established linear range do not need to be qualified.

**21.4.3.3.3** The linearity is verified at a frequency established by the manufacturer or method.

### **21.4.4 Calibration Verification**

The calibration relationship established during the initial calibration must be verified at periodic intervals 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, and is not appropriate nor permitted in SW-846 chromatographic procedures for trace environmental analyses.

- 21.4.4.1 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 or standard that can be injected within 12 hours of the beginning of the shift.
- 21.4.4.2 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 every 10 samples.
- 21.4.4.3 The acceptance limits for calibration verifications can be found in each method SOP. As a rule of thumb: GCMS  $\pm 20\%$ , GC and HPLC  $\pm 15\%$ , Inorganics:  $\pm 10$  or  $15\%$ . Actual methods may have wider or tighter limits (see the Method SOP for specifics).
- 21.4.4.4 If the response (or calculated concentration) for an analyte is within the acceptance limits of the response obtained during the initial calibration, then the initial calibration is considered still valid, and the analyst may continue to use the CF, RF or % drift values from the initial calibration to quantitate sample results.
- 21.4.4.5 If the response (or calculated concentration) for any analyte varies from the mean response obtained during the initial calibration by more than the acceptance criteria, then the initial calibration relationship may no longer be valid. If routine corrective action procedures fail to produce a second consecutive (immediate) calibration verification within acceptance criteria, then either the laboratory has to demonstrate performance after corrective action with two consecutive successful calibration verifications, or a new initial instrument calibration must be performed. However, sample data associated with an unacceptable calibration verification may be reported as qualified data under the following special conditions:
- 21.4.4.5.1** 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.
- 21.4.4.5.2** 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.

#### 21.4.4.6 Verification of Linear Calibrations

Calibration verification for linear 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. Use the equations below to calculate % Drift or % Difference, depending on the procedure specified in the method SOP. 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.

The Percent Difference is calculated as follows:

$$\% \text{ Difference} = \frac{(\text{CF}(v) \text{ or } \text{RF}(v)) - (\text{Avg. CF or RF})}{(\text{Avg. CF or RF})} \times 100$$

Where: CF(v) or RF(v) = CF or RF from verification standard  
Avg. CF or RF = Average CF or RF from Initial Calibration.

The Percent Drift is calculated as follows:

$$\% \text{ Drift} = \frac{\text{Result} - \text{True Value}}{\text{True Value}} \times 100$$

The Percent Recovery is calculated as follows:

$$\% \text{ Recovery} = \frac{\text{Result}}{\text{True Value}} \times 100$$

#### 21.4.4.7 Verification of a Non-Linear Calibration

Calibration verification of a non-linear calibration is performed using the percent drift or percent recovery calculations described in Section 21.4.4.6 above.

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.

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).

## **21.5 POLICY ON 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 will not be reported as a TIC. If the compound is reported on the same form as true TICs, it must 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 the following SOPs for guidelines for making tentative identifications (CORP-MS-0001NC, GC/MS Analysis Based on Method 8270C, and CORP-MS-0002NC, Determination of Volatile Organics by GC/MS based on Method 8260B & 8260A).

## **21.6 POLICY ON 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.

**21.6.1** The concentration of the BFB or DFTPP must be at or below the concentrations that are referenced in the analytical methods. Part of the purpose of the tune is to demonstrate sensitivity and analyzing solutions at higher concentrations does not support this purpose. Tune failures may be due to saturation and a lower BFB/DFTPP concentration may be warranted.

**21.6.2** Tune evaluations usually utilize the "Autofind" function and are set up to look at the apex +/- 1 scan and average the three scans. Background correction is required prior to the

start of the peak but no more than 20 scans before. Background correction cannot include any part of the target peak.

**21.6.3**     Other Options or if Auto Tune Fails:

21.6.3.1     Sometimes the instrument does not always correctly identify the apex on some peaks when the peak is not perfectly shaped. In this case, manually identify and average the apex peak +/- 1 scan and background correct as in 21.6.4 above. This is consistent with EPA 8260 and 8270.

21.6.3.2     Or the scan across the peak at one half peak height may be averaged and background corrected. This is consistent with Standard Methods 6200, EPA 624 and EPA 625.

21.6.3.3     Adjustments such as adjustments to the repeller and ion focus lenses, adjusting the EM Voltage, etc. may be made prior to tune verification as long as all of the subsequent injections in the 12-hour tune cycle are analyzed under the same MS tune settings and it is documented in the run sequence log and/or maintenance log that an adjustment was made. Excessive adjusting (more than two tries) without clear documentation is not allowed. Necessary maintenance is performed and documented in the Instrument Log.

21.6.3.4     A single scan at the Apex (only) may also be used for the evaluation of the tune. For SW 846 and EPA 600 series methods, background correction is still required.

21.6.3.5     Cleaning the source or other maintenance may be performed and then follow steps for tune evaluation above. Note: If significant maintenance was performed, see methods 8000B or 8000C then the instrument may require recalibration prior to proceeding.

**21.6.4**     Tune evaluation printouts must include the chromatogram and spectra as well as the Tune evaluation information. In addition, the verifications must be sent directly to the printer or pdf file (no screen prints for DFTPP or BFB tunes). This ability should be built into the instrument software.

**21.6.5**     All MS tune settings must remain constant between running the tune check and all other samples. It is recommended that a separate tune method not be used, however a separate method may be used as long as the MS conditions between the methods are the same as the sample analysis method and tracked so any changes that are made to the analysis method are also made to the tune method.

**Table 21-1.**

**Example - Laboratory Equipment and Instrumentation**

<b>Instrument Type</b>	<b>Manufacturer</b>	<b>Model</b>	<b>Auto-sampler</b>
Metals ICP	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
	Thermo Jarrell Ash	Trace Analyzer 61E	Yes
Metals ICP/MS	Perkin Elmer (I-7)	ELAN 6100	Yes
	Thermo (I-8)	Series 2	Yes
Metals Mercury Analyzer	Leeman (CVAA)	PS200 II	Yes
	Leeman (CVAF) Low Level	Hydra AF Gold+, Model #112-00067-1	Yes
	Leeman (CVAA)	Hydra AA	Yes
GC/MS Semivolatiles	Agilent A4AG2	5975-C	NA
	Hewlett-Packard HP7	5973-6890	NA
	Hewlett-Packard HP8	5973-6890	NA
	Hewlett-Packard HP9	5973-6890	NA
	Hewlett-Packard HP10	5973-6890	NA
GC/MS Volatiles	Hewlett-Packard (UX2)	5971A-5890	Yes
	Hewlett-Packard (HP6)	5973-6890	Yes
	Hewlett-Packard (UX7)	5973-6890	Yes
	Hewlett-Packard (UX8)	5973-6890	Yes
	Hewlett-Packard (UX9)	5973-6890	Yes
	Hewlett-Packard (UX10)	5973-6890	Yes
	Agilent (UX11) (former HP)	5973-6890	Yes
	Agilent (UX12)	5973-6890	Yes
	Agilent (UX14)	5973-6890	Yes
	Agilent (UX15)	5973-6890	Yes
	Agilent (UX16)	5975-6890	Yes
GC/MS Volatiles Autosampler	OI Analytical (UX2)	4552	
	OI Analytical (HP6)	4552	
	OI Analytical (UX7)	4552	
	OI Analytical (UX8)	4552	
	OI Analytical (UX9)	4552	
	OI Analytical (UX10)	4552	
	OI Analytical (UX11)	4552	
	OI Analytical (UX12)	4552	
	OI Analytical (UX14)	4552	
	OI Analytical (UX15)	4552	
	OI Analytical (UX16)	4552	

<b>Instrument Type</b>	<b>Manufacturer</b>	<b>Model</b>	<b>Auto-sampler</b>
GC/MS Volatiles Purge and Trap	OI Analytical (UX2)	4560	
	OI Analytical (HP6)	4560	
	OI Analytical (UX7)	4660	
	OI Analytical (UX8)	4560	
	OI Analytical (UX9)	4560	
	OI Analytical (UX10)	4560	
	OI Analytical (UX11)	4560	
	OI Analytical (UX12)	4560	
	OI Analytical (UX14)	4560	
	OI Analytical (UX15)	4660	
	OI Analytical (UX16)	4660	
	EST (spare)	Encon	
WC Ion Chromatograph	Dionex	DX-320	NA
	Dionex	DX-120	NA
WC TOC	OI Analytical	1010 TOC Analyzer	
WC TOX	Thermo Electron	1200-S/N 973515	
	Thermo Electron	1200-S/N 2001.0174	
	Thermo Electron	1200-S/N 2005.0234	
WC UV/VIS	Milton Roy	Spectronic 401	No
	Genesys	Spectronic 20	No
WC Autotitrator	Man-Tech	PC – Titrate	
WC pH Meter	Orion pH Meter	250A	
	Orion (Ammonia ISE)	520A	
WC Dissolved Oxygen Meter	YSI	52C E	No
WC Turbidimeter	HF Scientific	Micro 100	No
WC BOD	Labtronics, Inc.	BOD	
WC Block Digester	Andrews	2210 Phenol	
	Andrews	2205 Ammonia	
	Lachat	BD46 TKN	

<b>Instrument Type</b>	<b>Manufacturer</b>	<b>Model</b>	<b>Auto-sampler</b>
WC Cyanide	Midi Serial #1000-99-01	PRG-2520-BL	
WC Conductivity	Man-Tech	4310	
WC Flashpoint	Petrolab (199443)	Petrotest	
	Herzog	HFP 339	
WC Discrete Analyzer	Kone	Konelab 200	
	Kone	Konelab 250	
WC Residual Chlorine Meter	Hanna	HI 93701	
	Hanna	HI 93701	
WC TRAACS	Bran & Luebbe	800	
Ext. 6-Position Accelerated Soxhlet Extractor	Gerhardt Soxtherm	6 units	
Extractions Sonicators	Misonix	3000	NA
	Fisher	550	NA
GC Semivolatiles	Hewlett-Packard (P1)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P2)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P3)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P4)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P5)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P6)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P7)	6890 EPC & Dual FID	Yes
	Hewlett-Packard (P9)	6890 EPC & Dual ECD Y-Splitter	Yes
	Hewlett-Packard (P10)	6890 EPC & Dual ECD Y-Splitter	Yes

<b>Instrument Type</b>	<b>Manufacturer</b>	<b>Model</b>	<b>Auto-sampler</b>
GC Semivolatiles (Cont'd)	Hewlett-Packard (P11)	6890 EPC & Dual ECD Y-Splitter	Yes
GC Semivolatiles HPLC	Hewlett-Packard (L2)	HPLC 1100	Yes
	Hewlett-Packard (L3)	HPLC 1100	Yes
GC Volatiles	Agilent (A)	6890 PID/FID	Yes
	Hewlett-Packard (O)	6890 Dual PID/Hall	Yes
	Hewlett-Packard (P)	6890 PID/HALL	Yes
	Hewlett-Packard (Y)	6890N PID/FID	Yes
	Hewlett-Packard (P8)	6890 EPC & Dual FID	Yes
GC Volatiles Auto Sampler	OI Analytical (O)	Archon	
	OI Analytical (Y)	4552	
	Varian (A)	Archon	
	Varian (P)	4552	
GC Volatiles Purge & Trap	Tekmar (O)	4560	
	Tekmar (P)	3000	
	OI Analytical (A)	4560	
	OI Analytical (Y)	3000	
GM Bedford	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Agilent	6890N	Yes
	Misonix Sonicator	3000	NA

**Table 21-2.**

**Example – Laboratory Balance Inventory**

<b>Instrument</b>	<b>Location</b>	<b>Manufacturer</b>	<b>Model Number</b>	<b>Type</b>	<b>Serial Number</b>
B001	MS Volatiles	OHAUS	E400D	Top Loader	3317
B002	MS Volatiles	Mettler	AC 100	Analytical	B01177
B005	Metals	Sartarius	R300S	Analytical	38020045
B006	Wet Chemistry	American Scientific	SP180	Analytical	2904794
B007	High Hazard Room	American Scientific	ER-180A	Analytical	2904257
B008	Waste Building (no log book)	Howe Richardson	XL 5401		A029390
B009	Leachates (Extractions)	OHAUS	GT 4800	Top Loader	1687
B010	Wet Chemistry	Mettler	PJ 3600	Top Loader	G29475
B011	Extractions	American Scientific	SP 180	Analytical	2902127
B013	Extractions	OHAUS	TS 4KD	Top Loader	1936
B018	GC Volatiles	American Scientific	DTL 350	Analytical	10594
B019	GC Volatiles	Sartarius	L2200S	Top Loader	36020158
B022	Wet Chemistry	Mettler	PM300	Top Loader	F03040
B023	Wet Chemistry	OHAUS	TS 400S	Top Loader	3608
B024	Wet Chem Prep Room	Mettler	AE 100	Analytical	C25750
B025	Extractions	Mettler	PM 4600	Top Loader	G64548
B027	Wet Chemistry	American Scientific	SP 180	Analytical	2904154
B028	Wet Chemistry	Denver Instrument	M Series	Analytical	P119656
B030	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841870
B031	Metals (new 9/5/6)	AND	EK410i	Top Loader	P1841872
B032	Metals	OHAUS	ARC120	Top Loader	H28312
B033	Leachates (Extractions)	OHAUS	ARC120	Top Loader	H27612

Table 21-3.

**Example – Laboratory Refrigerators, Freezers, Ovens, Thermometers, Steambaths, Hotblocks, and Hotplates**

<b>Instrument Type</b>	<b>Manufacturer / Model</b>	<b>Location</b>	<b>Tracking No.</b>
Refrigerators	American Scientific CLP	BNA	R015
	VWR Scientific	BNA	R027
	Frigidaire	BNA	R031
	GE GPC	Extractions	R007
	Hotpoint “D”	Extractions	R008
	GE	Extractions	R010
	Hotpoint CLP Pest.	GC Semi	R016
	VWR	GC Semi	R021
	Frigidaire	GC Semi	R030
	Kelvinator	GC Semi	R032
	BB	GC VOA	R017
	True	MS Voa	R028
	Cryo Frig A CLP	MS Voa	R001
	Mr. Winter	MS Voa	R002
	#203 Enseco	MS Voa	R003
	Hotpoint “OO”	Org Haz	R012
	Kelvinator	Org Prep	R011
	Frigidaire	R&D	R013
	Sample Walk-In 2 <sup>nd</sup>	Sample Receiving	R018
	LaCross	Sample Receiving	R019
	Walk-In CLP	Sample Receiving	R020
	True	Sample Receiving	R025
	Baxter Cryo	Sample Receiving	R033
	True	Wet Chem	R024
	Frigidaire BOD	Wet Chem	R023
	Baxter Cryo Fridge BOD	Wet Chem	R029
	Frigidaire BOD	Wet Chem	R034
	Fisher Scientific 307 BOD	Wet Chem	R004
Ovens	TDS	Wet Chem	O-001
	WWR	Wet Chem	O-002
	TSS	Wet Chem	O-003
	TDS Evap	Wet Chem	O-004
	Lindberg/Blue	Wet Chem	O-006

Instrument Type	Manufacturer / Model	Location	Tracking No.
Freezers	Kenmore	BNA	F005
	Frigidaire	BNA	F012
	Hotpoint "D"	Extractions	F007
	Magic Chef	GC Semi	F004
	Marvel HPLC	GC Semi	F013
	GE	GC VO	F002
	Kenmore Side/Side	MS Voa	F001
	Frigidaire	MS Voa	F011
	Frigidaire	MS Voa	F016
	Kelvinator	Org Prep	F003
	Crosley	Receiving - Warehouse	F008
	GE	Receiving - Warehouse	F009
	Frigidaire	Receiving - Warehouse	F014
NIST Thermometers	Ertco S/N 69887 Product 15041D		92564
	Ertco S/N NB179010 Product N/A		167513
	Ertco S/N 3053 Product ASTM 068C-BF		15169121
	Ertco S/N 3243 Product ASTM 62C		163757
Steambaths		Extractions	A
		Extractions	B
		Extractions	C
Hotblocks		Metals	0
		Metals	1
		Metals	2
		Metals	3
		Metals	4
		Metals	A1
		Metals	A2
		Metals	B1
		Metals	C1
		Metals	C2
	Metals	C3	
	Metals	C4	
Hotplate		Metals	1-A
		Metals TCLP	9 K

**Table 21-4.**

**Example: Schedule of Routine Maintenance**

(Refer to manufacturer's instructions for each instrument to identify and perform maintenance operations)

**INSTRUMENT MAINTENANCE SCHEDULE  
 ION CHROMATOGRAPH**

<b>As Needed</b>	<b>Daily</b>	<b>Weekly</b>	<b>Monthly</b>
Clean micro-membrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.
Check fuses when power problems occur.	Check gases.	Check filter (inlet)	Check/change bed supports guard and analytical columns, if indicated.
Reactivate or change column when peak shape and resolution deteriorate or when retention time shortening indicates that exchange sites have become deactivated.	Check pump pressure.		
De-gas pump head when flow is erratic.	Check conductivity meter.		

**INSTRUMENT MAINTENANCE SCHEDULE  
 TOTAL ORGANIC HALIDE ANALYZER**

<b>Daily</b>	<b>As Needed</b>
Check electrodes for damage; polish the electrodes.	Examine and clean or replace pyrolysis tube.
Replace dehydrating fluid and electrolyte fluid.	Clean titration cell.
Clean quartz boat.	Observe gas flow.
Observe check valves during use for backfeed.	Replace reference electrode fluid.
At end of each day of use, wash out absorption module, empty electrolyte and fill cell with DI water. Empty dehydrator tube	Change quartz wool.
Perform cell performance check.	Replace O-rings and seals.

**INSTRUMENT MAINTENANCE SCHEDULE  
 HIGH PRESSURE LIQUID CHROMATOGRAPH**

<b>Daily</b>	<b>As Needed</b>
Check level of solution in reservoirs. If adding, verify that solvent is from the same source. If changing, rinse gas and delivery lines to prevent contamination of the new solvent.	Replace columns when peak shape and resolution indicate that chromatographic performance of column is below method requirements.
Check gas supply.	Oil autosampler slides when sample does not advance.
Flush with an appropriate solvent to remove all bubbles.	Rinse flow cell with 1N nitric acid if sensitivity low.
Pre-filter all samples.	Change pump seals when flow becomes inconsistent.
	Repack front end of column Backflush column.

**INSTRUMENT MAINTENANCE SCHEDULE  
 ICP AND ICP/MS**

<b>Daily</b>	<b>Monthly or As Needed</b>	<b>Semi-Annually</b>	<b>Annually</b>
Check vacuum pump gage. (<10 millitorr)	Clean plasma torch assembly to remove accumulated deposits	Change vacuum pump oil	Notify manufacturer service engineer for scheduled preventive maintenance service
Check cooling water supply system is full and drain bottle is not full. Also drain tubing is clear, tight fitting, and has few bends.	Clean nebulizer and drain chamber; keep free flowing to maintain optimum performance	Replace coolant water filter (may require more or less frequently depending on quality of water)	
Check nebulizer is not clogged	Clean filters on back of power unit to remove dust		
Check capillary tubing is clean and in good condition	Replace when needed: - peristaltic pump tubing - sample capillary tubing - autosampler sipper probe		
Check peristaltic pump windings are secure	- Check yttrium position - Check O-rings - Clean/lubricate pump rollers		
Check high voltage switch is on			
Check torch, glassware, aerosol injector tube, and bonnet are clean			

**INSTRUMENT MAINTENANCE SCHEDULE  
 CVAS AND CVAFS**

<b>Daily</b>	<b>As Needed</b>	<b>Annually</b>
Change drying tube	Change pump tubing	Change Hg lamp
Check pump tubing/drain tubing	Check/change Hg lamp	
Check gas pressure	Clean optical cell	
Check aperture reading	Lubricate pump	
Check tubing		

**INSTRUMENT MAINTENANCE SCHEDULE  
 GAS CHROMATOGRAPH**

<b>Daily *</b>	<b>As Needed</b>
Check for sufficient supply of carrier and detector gases. Check for correct column flow and/or inlet pressures.	Replace front portion of column packing or break off front portion of capillary columns. Replace column if this fails to restore column performance, or when column performance (e.g., peak tailing, poor resolution, high backgrounds, etc.) indicates it is required.  Quarterly FID: clean detector, only as needed—not quarterly/or semi-annually.
Check temperatures of injectors and detectors. Verify temperature programs by RT shift.	Change glass wool plug in injection port and/or replace injection port liner when front portion of column packing is changed or front portion of capillary column is removed.
Clean injector port weekly for TPH for 8015B, when breakdown fails; otherwise, when RT shift or bad samples run.	Annually FID: replace flame tip, only as needed.  Only as needed: ECD--detector cleaning and re-foiling, whenever loss of sensitivity, erratic response, or failing resolution is observed
Check baseline level during analysis of run—not maintenance.	Perform gas purity check (if high baseline indicates that impure carrier gas may be in use).

<p>Watched weekly: check reactor temperature of electrolytic conductivity detector.</p> <p>Inspect chromatogram to verify symmetrical peak shape and adequate resolution between closely eluting peaks, when analyzing pesticides; part of analysis—not maintenance.</p> <p>Clip column leader when chromatography looks bad—not daily.</p>	<p>Replace or repair flow controller if constant gas flow cannot be maintained.</p> <p>Replace fuse.</p> <p>Reactivate external carrier gas dryers.</p> <p>Detectors: clean when baseline indicates contamination or when response is low. FID: clean/replace jet, replace ignitor. ECD: follow manufacturer's suggested maintenance schedule.</p> <p>Reactivate flow controller filter dryers when presence of moisture is suspected.</p> <p>HP 7673 Autosampler: replace syringe, fill wash bottle, dispose of waste bottle contents.</p>
---	---

\*No daily maintenance done on any instrument/method. Weekly change IPL on TPH instrument. Everything else is on an "as needed" basis.

**INSTRUMENT MAINTENANCE SCHEDULE  
 MASS SPECTROMETER**

<b>Daily</b>	<b>Weekly</b>	<b>As Needed</b>	<b>Quarterly</b>	<b>Annually</b>
Check for sufficient gas supply. Check for correct column flow and/or inlet pressure.	Check mass calibration (PFTBA or FC-43)	Check level of oil in mechanical pumps and diffusion pump if vacuum is insufficient. Add oil if needed between maintenance.	Check ion source and analyzer (clean, replace parts as needed)	Replace the exhaust filters on the mechanical rough pump every 1-2 years.
Check temperatures of injector, detector. Verify temperature programs.		Replace electron multiplier when the tuning voltage approaches the maximum and/or when sensitivity falls below required levels.	Check vacuum, relays, gas pressures and flows	
Check inlets, septa		Clean Source, including all ceramics and lenses - the source cleaning is indicated by a variety of symptoms including inability of the analyst to tune the instrument to specifications, poor response, and high background contamination.	Change oil in the mechanical rough pump.	
Check baseline level		Repair/replace jet separator.		
Check values of lens voltages, electron multiplier, and relative abundance and mass assignments of the calibration compounds.		Replace filaments when both filaments burn out or performance indicates need for replacement.		

**INSTRUMENT MAINTENANCE SCHEDULE  
 TRAACS AUTO ANALYZER**

<b>As Needed</b>	<b>Daily</b>
Replaces air filter when progressive loss of air pressure is observed.	Check air pressure gauge (22 ± 2 psi)
Replace air valve tubing when occlusion in tubing is observed	Use recommended washout procedure (at end of analysis operations)
Change all pump tubes (or after 200 hours of pumping time, or after 1000 hours of pumping time)	
Clean sample probe shaft	
Replace pump platens	
Lightly lubricate the linear sample rails (use semi-fluid lubricant)	
Replace colorimeter lamp (or after 2500 hours of use)	

**INSTRUMENT MAINTENANCE SCHEDULE  
 ANALYTICAL/TOP LOADING BALANCES**

<b>Daily</b>	<b>Annually</b>
Check using Class 1-verified weights once daily or before use	Manufacturer cleaning and calibration
Clean pan and weighing compartment	

**INSTRUMENT MAINTENANCE SCHEDULE  
 REFRIGERATORS/WALK-IN COOLERS**

<b>Daily</b>	<b>As Needed</b>
Temperatures checked and logged	Refrigerant system and electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 OVENS**

<b>Daily</b>	<b>As Needed</b>
Temperatures checked and logged	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 SPECIFIC DIGITAL ION ANALYZER**

<b>Daily</b>	<b>As Needed</b>
Daily when used: <ul style="list-style-type: none"> <li>• Calibrate with check standards</li> <li>• Inspect electrode daily, clean as needed</li> <li>• Inspect electrode proper levels of filling solutions daily; fill as needed</li> <li>• Clean probe after each use</li> </ul>	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 TURBIDIMETER**

<b>Daily</b>	<b>Monthly</b>	<b>As Needed</b>
Daily when used: <ul style="list-style-type: none"> <li>• Adjust linearity on varying levels of NTU standards. Standardize with NTU standards</li> <li>• Inspect cells</li> </ul>	Clean instrument housing	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 DISSOLVED OXYGEN METER**

<b>Daily</b>	<b>As Needed</b>
Daily when used: <ul style="list-style-type: none"> <li>• Calibrate with saturated air</li> <li>• Check probe membrane for deterioration</li> <li>• Clean and replace membrane with HCl solution</li> </ul>	<ul style="list-style-type: none"> <li>• Electronics serviced</li> <li>• Clean and replace membrane with HCl solution</li> </ul>

**INSTRUMENT MAINTENANCE SCHEDULE  
 CONDUCTANCE METER**

<b>Daily</b>	<b>As Needed</b>
Daily when used: <ul style="list-style-type: none"> <li>• Check probe and cables</li> <li>• Inspect conductivity cell</li> </ul>	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 CHEMICAL OXYGEN DEMAND (COD) REACTOR<sup>1</sup>**

<b>Daily</b>	<b>As Needed</b>
Daily when used: <ul style="list-style-type: none"> <li>• Calibrate with check standards</li> </ul>	Electronics serviced

**INSTRUMENT MAINTENANCE SCHEDULE  
 SPECTROPHOTOMETER**

<b>As Needed</b>	<b>Daily</b>	<b>Monthly</b>	<b>Annually</b>
Dust the lamp and front of the front lens	Check the zero % adjustment	Clean windows	Check instrument manual
	Clean sample compartment		Perform wavelength calibration
	Clean cuvettes		Replace lamp annually or when erratic response is observed
			Clean and align optics

**INSTRUMENT MAINTENANCE SCHEDULE  
 pH METER**

<b>As Needed</b>	<b>Daily</b>
Clean electrode	Inspect electrode. Verify electrodes are properly connected and filled
Refill reference electrode	Inspect electrode proper levels of filling solutions. Make sure electrode is stored in buffer

**INSTRUMENT MAINTENANCE SCHEDULE  
 TOTAL ORGANIC CARBON ANALYZER**

<b>Daily</b>	<b>As Needed</b>	<b>Weekly</b>	<b>Monthly</b>
Check: <ul style="list-style-type: none"> <li>• Oxygen supply</li> <li>• Persulfate supply</li> <li>• Acid supply</li> <li>• Carrier gas flow rate (~ 150 cc/min)</li> <li>• IR millivolts for stability (after 30 min. warm-up)</li> <li>• Reagent reservoirs</li> </ul>	Check injection port septum after 50-200 runs  Tube end-fitting connections after 100 hours or use  Indicating drying tube NDIR zero, after 100 hours of use  Sample pump, after 2000 hours for use  Digestion vessel/condensation chamber, after 2000 hours of use  Permeation tube, after 2000 hours of use  NDIR cell, after 2000 hours of use  Change pump tubing	Check liquid-flow-rate-pump-tubing conditions on autosampler  Check injection port septum	Clean digestion vessel  Clean condenser column  Do the leak test

**Instrument Maintenance Schedule  
Digestion Block**

<b>Annually</b>
Check temperature with NIST thermometer

**Instrument Maintenance Schedule  
Flash Point Tester**

<b>Daily</b>
Check tubing Clean sample cup each use
Check gas
Clean flash assembly
Check stirrer

**Table 21-5.**

**Example: Periodic Equipment Calibrations**

<b>Type of Equipment</b>	<b>Calibration Requirements</b>
Balances	<ul style="list-style-type: none"> <li>• Must be serviced and calibrated annually by an approved vendor.</li> <li>• Calibration must be checked daily or before use by analyst with weight(s) classified as Class 1 (formerly termed Class S) by NIST or Class 1 traceable. Acceptance criteria vary according to weight used and accuracy of balance. Acceptance criteria must be documented in the log.</li> <li>• All Class 1 weights must be certified by an outside vendor every three years.</li> <li>• All non-Class 1 weights must be checked annually against NIST Class 1 weights annually.</li> </ul>
Thermometers	<ul style="list-style-type: none"> <li>• Working glass thermometers must be calibrated against a certified.</li> <li>• NIST thermometer at least annually as described in operation-specific SOPs.</li> <li>• Working non-glass thermometers must be calibrated against a certified NIST thermometer annually as described in operation-specific SOPs.</li> <li>• The NIST thermometer must be recertified every three years.</li> </ul>
Refrigerators/ Freezers	<ul style="list-style-type: none"> <li>• Thermometers must be immersed in a liquid such as mineral oil or glycol.</li> <li>• Temperature of units used for sample or standard storage must be checked daily as described in operation-specific SOPs. Refrigerator acceptance limits: <math>4^{\circ}\text{C} \pm 2^{\circ}\text{C}</math>. Freezer acceptance limits: <math>&lt; - 10^{\circ}\text{C}</math></li> </ul>
Ovens	<ul style="list-style-type: none"> <li>• Temperature of units must be checked daily or before use.</li> <li>• Acceptance limits vary according to use as described in operation-specific SOPs, and must be documented in the temperature log.</li> </ul>
Micropipettes	<ul style="list-style-type: none"> <li>• Calibrations are checked gravimetrically as required by the operation-specific SOP.</li> <li>• Must be calibrated at the frequency (normally quarterly) required by the manufacturer at a minimum.</li> </ul>
Syringes, Volumetric Glassware and Graduated Glassware	<ul style="list-style-type: none"> <li>• All syringes and volumetric glassware are purchased as Class A items.</li> <li>• Class A items are certified by the manufacturer to be within <math>\pm 1\%</math> of the measured volume; therefore, calibration of these items by TestAmerica laboratories is not required.</li> <li>• All analysts are trained in the proper use and maintenance of measuring devices to ensure the measurement of standards, reagents, and sample volumes are within method tolerances.</li> </ul>

**Table 21-6.**

**Example: Preventive Maintenance Procedures**

**SUMMARY OF INORGANIC METHOD CALIBRATIONS**

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Acidity	Initial	350.1	Two-level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	350.1	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	350.1	Third point check	9040B 9045C	Third point check
	Ending	350.1	One buffer check ± 5% of true value	9040B 9045C	N/A
Alkalinity, Bicarbonate, Carbonate	Initial	310.1 2320B	2 point calibration of pH meter ± 0.05 pH units of true value	--	N/A
	Continuing	310.1 2320B	N/A	--	N/A
	Ending	310.1 2320B	N/A	--	N/A
Ammonia	Initial	350.1	6 levels including blank, "r" <sup>3</sup> ≥ 0.995	--	N/A
	Continuing	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
	Ending	350.1	One level or LCS every 10 samples ± 10% of true value	--	N/A
Arsenic Speciation		N/A	N/A	7063	* Refer to Section 10 of SOP NC-WC-0090

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Biochemical Oxygen Demand (BOD)	Initial	405.1 SM5210B	a. Winkler titration: Iodometric with standard thiosulfate b. Membrane electrode: Read in air and in water with zero dissolved oxygen	--	N/A
	Continuing	405.1 SM5210B	N/A	--	N/A
	Ending	405.1 SM5210B	N/A	--	N/A
Bromide	Initial	300.0A	5 levels plus a blank, " $r^3 \geq 0.995$ "	9056A	5 levels plus a blank, " $r^3 \geq 0.995$ "
	Continuing	300.0A	Level every 10 samples $\pm 10\%$ of true value	9056A	N/A
	Ending	300.0A	N/A	9056A	N/A
Cation Exchange	Initial	N/A	N/A	9081	N/A
	Continuing	N/A	N/A	9081	N/A
	Ending	N/A	N/A	9081	N/A
Chemical Oxygen Demand (COD)	Initial	410.4 SM5220D	5 levels plus a blank " $r^3 \geq 0.995$ "	--	N/A
	Continuing	410.4 SM5220D	One level every 10 samples $\pm 10\%$ of true value	--	N/A
	Ending	410.4 SM5220D	One level $\pm 10\%$ of true value	--	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Chloride	Initial	300.0A 325.2	5 levels plus blank $"r" \geq 0.995$	9056A 9251	<u>Method 9056:</u> 3 levels plus a blank  <u>Method 9252:</u> 5 levels plus blank $"r" \geq 0.995$
	Continuing	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056:</u> One per batch of 20 samples, $\pm 10\%$ of true value  <u>Method 9252:</u> One level every 10 samples, $\pm 10\%$ of true value
	Ending	300.0A 325.2	One level every 10 samples $\pm 10\%$ of true value	9056A 9251	<u>Method 9056 :</u> N/A  <u>Method 9252:</u> One level $\pm 10\%$ of true value
Chromium Cr <sup>+6</sup>	Initial	3500 Cr-D	3 levels plus blank	7196A	5 levels plus blank $"r" \geq 0.995$
	Continuing	3500 Cr-D	One level every 10 samples  $\pm 10\%$ of true value	7196A	One level every 10 samples  $\pm 15\%$
	Ending	3500 Cr-D	One level $\pm 10\%$ of true value	7196A	One level $\pm 15\%$
Chlorine, Residual	Initial	330.5 SM3500CL-G	N/A	--	N/A
	Continuing	330.5 SM3500CL-G	N/A	--	N/A
	Ending	330.5 SM3500CL-G	N/A	--	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Conductivity	Initial	120.1 SM2510B	Standard KCl solution	9050A	One level to determine cell constant
	Continuing	120.1 SM2510B	N/A	9050A	N/A
	Ending	120.1 SM2510B	N/A	9050A	N/A
Cyanide (Amenable)	Initial	335.1 SM4500CN-G	6 levels plus blank "r" <sup>3</sup> ≥ 0.995	9012A	6 levels plus blank "r" <sup>3</sup> ≥ 0.995
	Continuing	335.1 SM4500CN-G	One level every 10 samples ± 10% of true	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1 SM4500CN-G	One level ± 10 % of true value	9012A	± 15% of true value
Cyanide (Total)	Initial	335.1/335.2 335.3/335.4 SM4500CU-E	6 levels plus blank "r" <sup>3</sup> ≥ 0.995	9012A	6 levels plus blank "r" <sup>3</sup> ≥ 0.995
	Continuing	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level every 10 samples ± 10 % of true value	9012A	One mid-level every 10 samples ± 15% of true value
	Ending	335.1/335.2 335.3/335.4 SM4500CU-E	One mid-level ± 10 % of true value	9012A	± 15% of true value
Flashpoint	Initial	--	N/A	1010 ASTM D93-9	p-Xylene reference standard must have flashpoint of 81°F ±2°F
	Continuing	--	N/A	1010 ASTM D93-9	N/A
	Ending	--	N/A	1010 ASTM D93-9	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Fluoride	Initial	300.0A 340.2	Method 300.0A: 5 levels plus a blank, "r" <sup>3</sup> ≥ 0.995  Method 340.2: 5 levels "r" <sup>3</sup> ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 340.2	One mid-level every 10 samples  ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 340.2	One mid-level ± 10% of true value	9056A	N/A
Hardness	Initial	130.2 2340B	Method 130.2: Standardize titrant  Method 2340B: See ICP Metals 200.7	--	N/A
	Continuing	130.2 2340B	Method 130.2: N/A  Method 2340B: See ICP Metals 200.7	--	N/A
	Ending	130.2 2340B	Method 130.2: N/A  Method 2340B: See ICP Metals 200.7	--	N/A
Iron (Ferrous)	Initial	3500-Fe D	3 levels plus a blank, "r" <sup>3</sup> ≥ 0.995	-	N/A
	Continuing	3500-Fe D	One mid-level every 10 samples ± 10% of true value	-	N/A
	Ending	3500-Fe D	One mid-level ± 10% of true value	-	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Nitrate	Initial	300.0A 353.2 SM4500NO <sub>3</sub> -E	5 levels plus a blank "r" <sup>3</sup> ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 353.2 SM4500NO <sub>3</sub> -E	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 353.2 SM4500NO <sub>3</sub> -E	One mid-level ± 10% of true value	9056A	N/A
Nitrite	Initial	300.0A 354.1	5 levels plus a blank "r" <sup>3</sup> ≥ 0.995	9056A	3 levels plus a blank
	Continuing	300.0A 354.1	One mid-level every 10 samples ± 10% of true value	9056A	One per batch of 20 samples ± 10% of true value
	Ending	300.0A 354.1	One mid-level ± 10% of true value	9056A	N/A
Nitrate-Nitrite	Initial	300.0A 353.2	5 levels plus blank "r" <sup>3</sup> ≥ 0.995	--	N/A
	Continuing	300.0A 353.2	One level every 10 samples ± 10% of true value	--	N/A
	Ending	300.0A 353.2	One mid-level ± 10% of true value	--	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Phosphorus (Total and Ortho-phosphate)	Initial	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: 3 levels plus a blank  Method 365.2: 5 levels plus a blank	--	N/A
	Continuing	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: One level every 10 samples ± 10% of true value  Method 365.2: Blank and 2 standards with each series of samples, ± 2% of true value or recalibrate	--	N/A
	Ending	300.0A 365.1 SM4500P-E	Method 300.0A/365.3: ± 10% of true value  Method 365.2: N/A	--	N/A
pH	Initial	150.1 SM4500H-B	2 level calibration that bracket the expected pH of the sample ± 0.05 pH units of true value	9040B 9045C	2 point calibration ± 0.05 pH units of true value
	Continuing	150.1 SM4500H-B	One buffer check every 10 samples ± 5% of true value	9040B 9045C	N/A
	Other	150.1 SM4500H-B	Third point check	9040B 9045C	Third point check
	Ending	150.1 SM4500H-B	One buffer check ± 5% of true value	9040B 9045C	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Phenolics	Initial	420.1	5 levels plus a blank "r" <sup>3</sup> ≥ 0.995	9065 9066	5 levels plus a blank "r" <sup>3</sup> 0.995
	Continuing	420.1	One mid-level every 10 samples ± 10% true value	9065 9066	One mid-level ± 15% true value
	Ending	420.1	One mid-level ± 10% true value	9065 9066	One mid-level ± 15% true value
Phosphate	Initial	SM4500P-E	N/A	9056	3 levels plus a blank
	Continuing	SM4500P-E	N/A	9056	One per batch of 20 samples ± 15% of true value
	Ending	SM4500P-E	N/A	9056	N/A
Settleable Solids	Initial	160.5	N/A	--	N/A
	Continuing	160.5	N/A	--	N/A
	Ending	160.5	N/A	--	N/A
Specific Conductance	Initial	120.1 SM2510B	Standardize meter with 0.01 M KCl	9050A	N/A
	Continuing	120.1 SM2510B	One level every 10 samples ± 10% of true value	9050A	N/A
	Ending	120.1 SM2510B	One level ± 10% of true value	9050A	N/A
Sulfate	Initial	300.0A 375.4	<u>Method 300.0A:</u> 5 levels plus blank "r" <sup>3</sup> ≥ 0.995  <u>Method 375.4:</u> 3 levels plus blank "r" <sup>3</sup> ≥ 0.995	9038 9056A	<u>Method 9038:</u> 3 levels plus a blank for every hour of continuous sample analysis.  <u>Method 9056:</u> 3 levels plus a blank

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Sulfate (Cont'd)	Continuing	300.0A 375.4	<u>Method 300.0A:</u> One mid-level after every 10 samples ± 10% of true value  <u>Method 375.4:</u> One level every 3 or 4 samples ± 10% of true value	9038 9056A	<u>Method 9038:</u> Independent-prepared check standard every 15 samples  <u>Method 9056:</u> 1 per batch of 20 samples ± 10% of true value
	Ending	300.0A 375.4	± 10% of true value	9038 9056A	N/A
Sulfide	Initial	376.1	<u>Method 376.1:</u> This is a titration method. Therefore, calibrations are not applicable.	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Continuing	376.1	<u>Method 376.1:</u> N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
	Ending	376.1	<u>Method 376.1:</u> N/A	9030B/ 9034 9030A	This is a colorimetric titration. Therefore, calibration is not applicable.
Total Dissolved Solids	Initial	160.1 SM2540E	This is a gravimetric determination. Calibrate balance prior to analysis	--	N/A
	Continuing	160.1 SM2540E		--	N/A
	Ending	160.1 SM2540E		--	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Total Kjeldahl Nitrogen (TKN)	Initial	351.3 SM4500NO <sub>3</sub>	Method 351.3: Titrimetric: Standardize titrant Colorimetric: 7 levels plus blank	--	N/A
	Continuing	351.3 SM4500NO <sub>3</sub>	Method 351.3: N/A	--	N/A
	Ending	351.3 SM4500NO <sub>3</sub>	Method 351.3: N/A	--	N/A
Total Organic Carbon (TOC)	Initial	415.1 SM5310D	3 levels plus blank	9060 Walkley Black	3 levels plus blank "r" <sup>3</sup> ≥ 0.995
	Continuing	415.1 SM5310D	1 mid-level every 10 samples ± 15% of true value	9060 Walkley Black	1 mid-level every 10 samples ± 15% of true value
	Ending	415.1 SM5310D	± 15% of true value	9060 Walkley Black	± 15% of true value
Total Organic Halides (TOX)	Initial	450.1	Method 450.1: Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently- prepared check standard	9020B 9023 (EOX)	Daily instrument calibration standard and blank in duplicate ± 10% of true value (calibration standard) Verify with independently-prepared check standard –ICV ± 10% SOP CORP-WC- 0001

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Total Organic Halides (TOX) (cont'd)	Continuing	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
	Ending	450.1	± 10% of true value	9020B 9023 (EOX)	CCV ± 10% of true value SOP CORP-WC-0001
Total Solids	Initial	160.3	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.3		--	N/A
	Ending	160.3		--	N/A
Total Suspended Solids (Nonfilterable)	Initial	160.2 SM2540E	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.2 SM2540E		--	N/A
	Ending	160.2 SM2540E		--	N/A
Turbidity	Initial	180.1	Minimum of 1 level in each instrument range. Follow manufacturer's instructions	--	N/A
	Continuing	180.1	N/A	--	N/A
	Ending	180.1	N/A	--	N/A
Volatile Solids	Initial	160.4	This is a gravimetric determination. Calibrate balance before use.	--	N/A
	Continuing	160.4		--	N/A
	Ending	160.4		--	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
ICP & ICP/MS Metals (excludes Hg)	Initial	200.7	One level and blank. ICV RSD <3% from replicate	6010B	One level and blank. ICV RSD <5% from replicate
	Initial	200.8	One level and blank	6020	One level and blank
	Continuing	200.7	Every 10 samples ±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard Every 10 samples ± 10% of true value CCV RSD < 5% from replicate
	Continuing	200.8	N/A	6020	N/A
	Ending	200.7	±10% of true value CCV RSD < 5% from replicate	6010B	Mid-level calibration standard ± 10% of true value CCV RSD < 5% from replicate
	Ending	200.8	N/A	6020	N/A
	Other	200.7	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP  Semi-Annually: ICP interelement correction factors Instrument detection limits	6010B	ICSA, ICSAB: Analyze at beginning of run. For ICSA, AB criteria see SOP  Semi-Annually: ICP interelement correction factors Instrument detection limits
	Other	200.8	N/A	6020	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Mercury by CVAA & CVAFS	Initial	245.1 1631E	5 levels plus blank ICV $\pm 10\%$ of true value " $r$ " <sup>3</sup> $\geq 0.995$	7470A 7471A	5 levels plus blank ICV $\pm 10\%$ of true value " $r$ " <sup>3</sup> $\geq 0.995$
	Continuing	245.1* 1631E	Daily or every 10 samples, whichever is more frequent $\pm 20\%$ of true value	7470A 7471A	Every 10 samples $\pm 20\%$ of true value
	Ending	245.1 1631E	$\pm 20\%$ of true value	7470A 7471A	$\pm 20\%$ of original prepared standard
	Other	245.1 1631E	<u>Annually:</u> MDL	7470A 7471A	<u>Annually:</u> MDL

\* 245.1 continuing – Initial CCV  $\pm 5\%$  of true value

**Footnotes**

- <sup>1</sup> National Pollutant Discharge Elimination System.
- <sup>2</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December, 1996).
- <sup>3</sup> " $r$ " = correlation coefficient.

**SUMMARY OF ORGANIC METHOD CALIBRATIONS**

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Aromatic Volatiles by GC	Initial	602	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	602	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections or 12 hrs whichever is greater, % D ≤ 15%, gases 20% D. Evaluate per SOP
	Ending	602	Run closer per NELAC requirement, but no criteria	8021B	Mid-level calibration standard, % D ≤ 15%. Evaluate per SOP, except as noted above.
	Other	602	N/A	8021B	N/A
Herbicides by GC	Initial	615 <sup>9</sup>	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8151A	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	615 <sup>9</sup>	One or more calibration standards analyzed daily  % D ± 15% of predicted response	8151A	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	615 <sup>9</sup>	N/A	8151A	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	615 <sup>9</sup>	N/A	8151A	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Pesticides/ PCBs by GC	Initial	608	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed	8081A 8082	Minimum of 5 levels. If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. (See SOP CORP-GC-0001)
	Continuing	608	One or more  calibration  standards analyzed  daily.  % D ± 15% of predicted response	8081A 8082	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	608	N/A	8081A 8082	Mid-level calibration standard. % D < 15% of predicted response for any analyte quantitated and reported.
	Other	608	N/A	8081A 8082	N/A
Petroleum Hydrocarbons/ Oil and Grease	Initial	1664A	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance	9071B	Calibrate analytical balance at 2 mg and 1000 mg Calibration must be ± 10% at 2 mg and ± 0.5% at 1000 mg or recalibrate balance
	Continuing	1664A	N/A	9071B	N/A
	Ending	1664A	N/A	9071B	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Purgeable Halocarbons by GC	Initial	601	Minimum of 3 levels If % RSD < 10%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed. Six levels for quadratic equation.
	Continuing	601	Analyze QC check sample and evaluate per method requirements	8021B	Mid-level calibration standard analyzed every 10 injections, or 12 hrs, whichever is greater. % D < 15%, gases 20% D Evaluate per SOP
	Ending	601	Run per NELAC, but no requirement to meet	8021B	Mid-level calibration standard, % D <15%.  Evaluate per SOP
	Other	601	N/A	8021B	N/A
Halogenated Volatiles by GC	Initial	--	N/A	8021B	Minimum of 5 levels If % RSD < 20%, use avg RF. Otherwise, calibration curve employed.
	Continuing	--	N/A	8021B	Mid-level calibration standard analyzed every 10 samples. % D < 15% of predicted response for any analyte quantitated and reported.
	Ending	--	N/A	8021B	Mid-level calibration standard % D < 15% of predicted response for any analyte quantitated and reported.
	Other	--	N/A	8021B	N/A

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Semivolatiles	Initial	625	Minimum of 3 levels, lowest near but above MDL. If % RSD $\leq$ 35%, use avg RF. Otherwise calibration curve employed.	8270C	Minimum of 5 levels, % RSD for RF for CCCs <sup>(4)</sup> < 30% SPCCs <sup>(5)</sup> : RF > 0.050
	Continuing	625	One level every 24 ours. Acceptance criteria are found in the method and SOP.	8270C	Mid-level standard every 12 hours (after tuning) %D for CCCs <sup>(4)</sup> < 20 % between RF from standard and avg RF from initial SPCCs <sup>(5)</sup> : RF > 0.050.
	Ending	625	N/A	8270C	N/A
	Other	625	DFTPP <sup>(7)</sup> tuning every 24 hours before standard or sample runs.	8270C	DFTPP <sup>(7)</sup> tuning at the beginning of every 12 hour shift.

Analysis	Calibration	NPDES <sup>1</sup>		RCRA (SW846) <sup>2</sup>	
		Method	Requirement	Method	Requirement
Volatiles	Initial	624	Minimum of 3 levels, lowest near but above MDL. If % RSD ≤ 35%, use avg RF. Otherwise, calibration curve employed.	8260B	Minimum of 5 levels, %RSD for RF for CCCs <sup>4</sup> < 30.0% SPCCs <sup>5</sup> : RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and  RF > 0.100 for Bromoform
	Continuing	624	1 level every 24 hours  Acceptance criteria are found in the method and SOP	8260B	Mid-level standard every 12 hours (after tuning)  %Drift for CCCs <sup>(4)</sup> < 20.0% between RF from standard and avg RF from initial  SPCCs <sup>(5)</sup> : RF ≥ 0.300 for Chlorobenzene and 1,1,2,2-tetrachloroethane, Chloromethane and 1,1-dichloroethane, and RF > 0.100 for Bromoform
	Ending	624	N/A	8260B	N/A
	Other	624	BFB <sup>(6)</sup> tuning at the beginning of every 24 hour shift.	8260B	BFB <sup>(6)</sup> tuning at the beginning of every 12 hour shift.

**Footnotes:**

- <sup>1</sup> National Pollutant Discharge Elimination System.
- <sup>2</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- <sup>3</sup> TCDD - 2,3,7,8-Tetrachlorodibenzo-p-dioxin.
- <sup>4</sup> CCC - Continuing Calibration Compounds.
- <sup>5</sup> SPCC - System Performance Check Compound.
- <sup>6</sup> BFB – Bromofluorobenzene.
- <sup>7</sup> DFTPP – Decafluorotriphenylphosphine.
- <sup>8</sup> Footnote deleted.
- <sup>9</sup> Method not listed in 40 CFR Part 136.

## 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. 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 six months of use. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. The following definitions are provided by the American Association for Laboratory Accreditation (A2LA):

“Traceability is the property of a measurement result whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, each step in the chain having stated uncertainties.” There are six essential elements:

- An unbroken chain of comparison
- A calculated measurement uncertainty for each step in the chain to allow for an overall uncertainty calculation
- Documentation of each step in each calibration report
- All steps in the chain are performed by individuals with evidence of technical competence and accredited by a recognized accreditation body
- Reference to International Standard (SI) units
- Recalibration at appropriate intervals to preserve traceability

Calibration is defined as “determining and documenting the deviation of the indication of a measuring instrument (or the stated value of a material measure) from the conventional ‘true’ value of the measurand.”

Uncertainty is defined as “a parameter associated with the result of a measurement that characterizes the dispersion of the value that could reasonably be attributed to the measurand.” Measurement of Uncertainty is discussed in Section 20 of this QA Manual.

## **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. Refer to Section 21 for calibration of weights and thermometers.

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.

## **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, 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. (Refer to Section 9 for additional information on purchasing). 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, 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 Table 9-1 in Section 9 for general storage requirements and Table 22-1 for additional storage information. Please refer to SOP NC-QA-0017, Standards and Reagents, for additional details. 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 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 in each group. 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.

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 standard or reagent logbooks, and are assigned a unique identification number. The following information is typically recorded in the Standards Logbook.

- 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 box (text field)

Records are maintained in each group 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
- Standard ID (from the Standards 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
- 3) According to Table 22-1

## **SECTION 23.0**

### **SAMPLING (NELAC 5.5.7)**

#### **23.1 OVERVIEW**

TestAmerica North Canton provides sampling services. Sampling procedures are described in SOP NC-SC-0006, Sample Procurement Protocol.

#### **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 available from the vendor electronically, or stored 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 Bisulfate – ACS Grade or equivalent
- Sodium Hydroxide – Instra-Analyzed or equivalent
- Sulfuric Acid – Instra-Analyzed or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent

##### **23.2.2 Preparing Container Orders**

Container orders are prepared using the laboratory Shipping Department, SOP NC-QA-0012. The laboratory also provides EnCore, TerraCore or other soil sampling devices when requested.

If containers are provided directly to the client from the manufacturer or from other sources, the laboratory will not be responsible for any of the above records.

#### **23.3 FIELD QUALITY CONTROL (QC)**

Common field quality control samples are defined in the following paragraphs. The frequency of field quality control samples should be specified in the site specific Quality Assurance Project Plan (QAPP) or by the client. TestAmerica provides trip blanks for VOC analysis with the sample

containers for all volatile organic analyses. Blanks generated in the field will be analyzed along with the field samples (exception soil samples where the blank is aqueous).

**23.3.1** Equipment Blank / Rinsate Blank - The equipment blank, sometimes referred to as a rinsate blank, is a sample of the water used to decontaminate sampling equipment. The source water should be as free of target analytes as possible. An aliquot of this water is poured over or through the sample collection device after decontamination, collected in a sample container, preserved with appropriate reagents, and returned to the laboratory. This serves as a check on sampling device cleanliness, and will also be affected by the site and sample handling conditions evaluated by the other types of blanks. The sampling time for the equipment blank should begin when the equipment is rinsed and the water is collected. Equipment blank collection for low level mercury is generally done at a clean facility rather than at the sample collection site.

**23.3.2** Field Blank - The field blank is water that is as free of target analytes as possible and from the same source as the equipment blank. The water is poured into a sampling container at the sampling site, preserved with the appropriate reagents, and returned to the laboratory. This serves as a check on reagent and environmental contamination. The sampling time for the field blank should be when the blank is prepared in the field.

**23.3.3** Trip Blank - The trip blank pertains to volatile analysis only. This serves as a check on sample contamination originating from sample transport, sample container contamination, shipping and storage, or from certain site conditions. Trip blanks are often referred to as travel blanks. They are prepared using pre-cleaned sample containers. They are filled with organic-free water (the source of the organic free water is the same source of water used to prepare volatile standards, method blanks, LCS and sample dilutions), sealed and taken into the field with the empty containers which will be used for sampling. The recommended frequency is one trip blank per cooler (in duplicate or triplicate), per volatiles method. Unless otherwise specified, the sampling time for the trip blank is the time of receipt at the laboratory (When the "Trip" ends).

**23.3.4** Field Duplicates - Field duplicates are replicate samples collected from the same sampling point or location during a field collection event. This control sample is used to demonstrate the ability of both the sampling and analytical process to generate data of acceptable precision.

## **23.4** 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 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.

**23.4.1** Semi-Volatile - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day (and time where applicable) solvent contacts the sample. If a sample is to be extracted on the day of expiration, the actual time of extraction must be recorded on the sample preparation worksheet. Holding

times for analysis are measured from the date (and time where applicable) of initiation of extraction to the time of injection into the gas chromatograph.

**23.4.2** Volatiles - Holding times for volatile organics are measured from the date (and time where applicable) of sampling to the date and time of injection into the gas chromatograph.

**23.4.3** Inorganics - For inorganic and metals analysis, the preparation/digestion/distillation must be started within the maximum holding time as measured from the sampling date (and time where applicable).

### **23.5** 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 (refer to Tables 23-1 to 23-7) 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.

### **23.6** 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. In that regard the following guidelines apply to analysts:

Analysts must 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.

**23.6.1** For water samples, before taking each aliquot for analysis, invert the sample container end-over-end three times and immediately pour off the aliquot. Especially when suspended solids are present, adequate mixing of the sample is extremely important.

**23.6.2** For solid samples, when volatile organics are not requested, if the solid can be mixed, stir before removing the aliquot. When possible stir the entire sample in the sample container. Remove small increments of sample from different parts of the container to make up the complete subsample.

- For soil samples, avoid debris in the subsample aliquot as much as possible (e.g. gravel, sticks, roots and grass); note this information in the sample preparation record.

**23.6.3** For solid samples, when volatile organics analysis is requested, the sample should be manipulated as little as possible. In most cases, the sample will arrive already preserved or in an EnCore™ sampler of the correct mass (requiring quick preservation of the entire amount). If the client requests volatiles on a solid sample which has been collected in a jar and is in a common container from which aliquots for other test methods must be taken, login should deliver the container to the volatiles department for preparing a proper aliquot prior to any other aliquots being taken out.

**23.6.4** For multiphasic samples, the client should instruct the laboratory as to the intent of the testing and how to handle the sample. If the entire sample is to be accounted for, and the phases do not mix easily with inversion/stirring, such that a representative aliquot can be taken, the analyst must record the percent by volume of each phase. The analysis must be conducted on each phase separately; the final results are combined mathematically, weighting the individual phase results by volume. One exception to this procedure is the situation addressed in the TCLP and SPLP methods for wastes containing free liquids. However, if the leachate and final filtrate are not miscible, it is necessary to combine mathematically the concentrations of the two (or more) solutions by volume. A laboratory coliwasa can be used to subsample a multilayer liquid sample when appropriate.

Tables 23-1 to 23-7 detail holding times, preservation and container requirements, and sample volumes for SDWA and NPDES methods. The sample volumes are intended to be a minimal amount to perform the method. The containers used may be of larger size. **Please note:** The holding times are program specific and different programs may have different holding times for equivalent methods, e.g., there are differences in holding times for many organic analytes between SDWA and NPDES. RCRA methods may also be different.

Table 23-1.

Inorganic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Acidity	Water	100 mL	305.1 SM2310B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
Alkalinity, Bicarbonate, Carbonate	Water	100 mL	310.1 2320B	250 mL plastic or glass. Cool to 4°C, 14 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ammonia	Water	100 mL	350.1 350.2 SM4500NH <sub>3</sub> -E SM4500NH <sub>3</sub> -F	500 mL plastic or glass. Cool to 4°C H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Arsenic (ASV) Anodic Stripping Voltammetry	Water	100 mL	---	N/A	7063	250 mL plastic. Cool to 4°C. HCl to pH < 2, 28 days
Biochemical Oxygen Demand (BOD), Carbonaceous	Water	1000 mL	405.1 5210B	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Bromide	Water	50 mL	300.0A <sup>7</sup>	250 mL plastic or glass. No preservative required, 28 days	9056A	Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Cation Exchange	Solid	8 oz	---	N/A	9081	8 or 16 oz glass. Cool to 4°C, 6 months
Chemical Oxygen Demand (COD)	Water	100 mL	410.4 5220D	250 mL glass or plastic. Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Chloride	Water	50 mL	300.0A <sup>7</sup> 325.2	250 mL plastic or glass. No preservative required, 28 days	9056A 9251	Method 9056: Cool to 4°C. Analyze ASAP after collection. Method 9251: 250ml plastic or glass, no preservative required, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chlorine, Residual	Water	100 mL	330.5	250 mL glass or plastic. Cool to 4°C, analyze immediately	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Chromium (Cr <sup>+6</sup> )	Water	100 mL	3500 Cr-D	Method 218.4: 200 mL plastic or glass. Cool to 4°C, 24 hours. Method 3500 Cr-D: 200 mL quartz, TFE, or polypropylene HNO <sub>3</sub> to pH <2. Cool to 4°C. Analyze ASAP after collection	7196A	200 mL plastic or glass. Cool to 4°C, 24 hours
	Solid	20 g	---	N/A	7196A 3060A	250 mL plastic or glass, 30 days to digestion, 96 hours after digestion
	Waste	N/A	---	N/A	---	N/A
Conductivity	Water	100 mL	120.1 2510B	200 mL glass or plastic. Cool to 4°C, 28 days	9050A	200 mL glass or plastic. Cool to 4°C, 24 hours
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Cyanide (Amenable)	Water	250 mL	335.1 SM4500CN-G	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid <sup>6</sup> . Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid <sup>6</sup> . Cool to 4°C, 14 days
	Solid	50g	---	N/A	9012A	Not Specified
	Waste	50g	---	N/A	9012A	Not Specified
Cyanide (Total)	Water	1L	335.2 335.3 335.4 <sup>(7)</sup> SM4500CN-E	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid <sup>6</sup> . Cool to 4°C, 14 days unless sulfide is present. Then maximum holding time is 24 hours.	9012A	1 liter plastic or glass, NaOH to pH >12 0.6g ascorbic acid <sup>6</sup> . Cool to 4°C, 14 days.
	Solid	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C, 14 days
	Waste	50g	--	N/A	9012A	8 or 16 oz glass Teflon-lined lids, Cool to 4°C
Flashpoint (Ignitability)	Liquid	100 mL	---	N/A	1010 ASTM D93-9	No requirements, 250 mL amber glass. Cool to 4°C recommended
	Solid	100 g	--	N/A	---	N/A
	Waste	100 mL	--	N/A	---	N/A
Fluoride	Water	300 mL	300.0 / 340.2	500 mL plastic. No preservation required, 28 days.	9056A	Cool to 4°C. Analyze ASAP after collection.
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Hardness (Total)	Water	50 mL	130.2 2340B	250 mL glass or plastic, HNO <sub>3</sub> to pH < 2, 6 months	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Iron (Ferrous)	Water	100 mL	3500-Fe D	1 liter glass or polyethylene container, 6 months. <b>This test should be performed in the field.</b>	-	N/A
	Solid	N/A	-	N/A	-	N/A
	Waste	N/A	-	N/A	-	N/A
Nitrate	Water	50 mL	300.0A <sup>7</sup> SM4500NO <sub>3</sub> - E	Method 300.0: 250 mL plastic or glass. Cool to 4°C, 48 hours.	9056A	Method 9056: Cool to 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	Not Specified
Nitrite	Water	50 mL	300.0A <sup>7</sup> 354.1	250 mL plastic or glass. Cool, 4°C, 48 hours	9056A	Cool, 4°C. Analyze ASAP after collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Nitrate-Nitrite	Water	50 mL	353.2	250 mL plastic or glass, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Ortho-phosphate	Water	50 mL	300.0A <sup>7</sup> 365.1 SM4500P-E	100 mL plastic or glass. Filter on site.  Cool to 4°C, 48 hours	9056A	Cool to 4°C. Analyze ASAP collection
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
pH	Water	50 mL	150.1 SM4500H-B	100 mL plastic or glass. Analyze immediately. <b>This test should be performed in the field.</b>	9040B	100 mL plastic or glass. Analyze immediately. <b>This test should be performed in the field.</b> <sup>(8)</sup>
	Solid	N/A	---	N/A	9045C	4 oz glass or plastic. Cool to 4°C. Analyze as soon as possible. <sup>8</sup>
	Waste	N/A	---	N/A	9045C	4 oz glass or plastic, Cool to 4°C. Analyze as soon as possible. <sup>8</sup>
Phenolics	Water	100 mL	420.1	500 mL glass, Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	9065	1 liter glass recommended, Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 4, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	9065	Not Specified
Phosphate	Water	50 mL	---	N/A	9056A	Cool to 4°C, analyze ASAP collection
	Solid	N/A	---	N/A	9056A	N/A
	Waste	N/A	---	N/A	9056A	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Phosphorus (Total)	Water	100 mL	365.2 365.1 SM4500P-E	100 mL plastic or glass, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Settleable Solids	Water	1000 mL	160.5	1000 mL plastic or glass. Cool to 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Specific Conductance	Water	50 mL	120.1 2510B	250 mL plastic or glass. Cool to 4°C, 24 hours	9050A	250 mL plastic or glass. Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Sulfate (SO <sub>4</sub> )	Water	50 mL	300.0A <sup>7</sup> 375.4	100 mL plastic or glass. Cool to 4°C, 28 days	9056A 9038	Method 9056: Cool to 4°C. Analyze ASAP collection. Method 9038: 200 mL plastic or glass, Cool to 4°C, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	100 mL	---	N/A	9038	200 mL plastic or glass. Cool to 4°C, 28 days

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Sulfide	Water	250 mL	376.1	500 mL plastic or glass. Cool to 4°C, Add 2 mL zinc acetate plus NaOH to pH > 9, 7 days	9030A 9030B/ 9034	500 mL plastic, No headspace. Cool to 4°C. Add 4 drops of 2N zinc acetate per 100 mL of sample, adjust the pH to > 9 with 6 N NaOH solution, 7 days
	Solid	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
	Waste	50 g	---	N/A	9030A 9030B/ 9034	Cool to 4°C. Fill surface of solid with 2N Zinc acetate until moistened. Store headspace-free
Total Dissolved Solids (Filterable)	Water	100 mL	160.1 2540C	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Total Kjeldahl Nitrogen (TKN)	Water	100 mL	351.3	500 mL plastic or glass. Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Organic Carbon (TOC)	Water	100 mL	415.1 SM5310D	100 mL plastic or glass. Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2, 28 days	9060 Walkley-Black	100 mL glass or 40 mL VOA vials, Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> or HCl to pH < 2, 28 days
	Solid	N/A	---	N/A	9060 Walkley-Black	Not Specified
	Waste	N/A	---	N/A	9060 Walkley-Black	Not Specified
Total Organic Halides (TOX) (EOX)	Water	100 mL	450.1 <sup>(7)</sup>	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, HNO <sub>3</sub> to pH < 2, no headspace, 28 days	9020B  9023 (EOX)	500 mL amber glass, Teflon®-lined lid. Cool to 4°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2, no headspace, 28 days
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Solids	Water	100 mL	160.3	250 mL plastic or glass. Cool to 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Total Suspended Solids (Nonfilterable)	Water	100 mL	160.2	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3, 7</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method	Requirements
Turbidity	Water	50 mL	180.1	250 mL plastic or glass. Cool, 4°C, 48 hours	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Volatile Solids	Water	100 mL	160.4	250 mL plastic or glass. Cool, 4°C, 7 days	---	N/A
	Solid	N/A	---	N/A	---	N/A
	Waste	N/A	---	N/A	---	N/A
Metals (excludes Hg)	Water	100 mL	200 series	1 liter glass or polyethylene container, HNO <sub>3</sub> to pH ≤ 2, 6 months	6010B 6020	1 liter glass or polyethylene container, HNO <sub>3</sub> to pH ≤ 2, 6 months
	Solid	200 g	200 series	2, 8, or 16 oz glass or polyethylene container storage at 4 °C	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
	Waste	200 g	200 series	N/A	6010B 6020	8 or 16 oz glass or polyethylene container, storage at 4°C, 6 months
Mercury (CVAA) (CVAFS)	Water	100 mL	245.1 245.7 1631E	250 mL glass or polyethylene container, HNO <sub>3</sub> to pH ≤ 2, 28 days	7470A	1 liter glass or polyethylene container, HNO <sub>3</sub> to pH ≤ 2, 28 days
	Solid	200 g	--	2, 8, or 16 oz glass or polyethylene container. Cool to 4°C, 28 days. Not applicable for Method 1631E.	7471A	8 or 16 oz glass or polyethylene container. Cool to 4°C, 28 days (CORP-MT-0007)
	Waste	200 g	--	N/A	7471A	8 or 16 oz glass or polyethylene container. Cool, 4°C, 28 days (CORP-MT-0007)

**Footnotes**

- <sup>1</sup> Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
- <sup>2</sup> National Pollutant Discharge Elimination System - MCAWW, March 1983.
- <sup>3</sup> Holding times are calculated from date of collection.
- <sup>4</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, (SW-846), Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA, (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- <sup>5</sup> Solid matrix type includes soil, sediment, sludge and other solid materials not classified as waste.
- <sup>6</sup> Samples to be analyzed for cyanide should be field-tested for residual chlorine. If residual chlorine is detected, ascorbic acid should be added.
- <sup>7</sup> Method not listed in 40 CFR Part 136.
- <sup>8</sup> If not done in the field (ASAP) per the method and requested by client, analyze in lab within 48 hours.
- <sup>9</sup> EPA issued memo recommending not to use reactive cyanide and sulfide methods.

Table 23-2.

Organic Sample Containers, Preservatives, and Holding Times

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Aromatic Volatiles	Water	40 mL	602	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 7 days with pH > 2, 14 days with pH ≤ 2	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH < 2, 14 days with pH ≤ 2
	Solid <sup>5</sup>	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C <sup>12</sup>
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Max holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool to 4°C <sup>12</sup>

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2,3</sup>		RCRA (SW846) <sup>3,4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Halogenated Volatiles by GC	Water	40 mL	--	N/A	8021B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C, 1:1 HCl to pH ≤ 2, 14 days
	Solid <sup>5</sup>	5 g or 25 g	--		8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool to 4°C <sup>12</sup>
	Waste	5 g or 25 g	--	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore Sampler is 48 hours (before the sample is added to methanol). Cool, 4°C <sup>12</sup> .

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Herbicides	Water	1L	615 <sup>(10)</sup>	1 liter amber glass with Teflon®-lined lid, Sodium thiosulfate or ascorbic acid if residual chlorine present. Cool to 4°C, Extraction, 7 days. Analysis, 40 days after extraction	8151A	1 liter amber glass with Teflon®-lined lid. If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool to 4°C. Extraction, 7 days. Analysis, 40 days of the start of extraction.
	Solid	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	--	N/A	8151A	4 or 8 oz glass widemouth with Teflon®-lined lid. Cool to 4 °C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Pesticides/ PCBs	Water	1L	608	1 liter amber glass with Teflon®-lined lid, Adjust pH to 5-9 if extraction not to be done within 72 hours of sampling. Add sodium thiosulfate if residual chlorine present and aldrin is being determined. Cool, 4°C. Extraction, 7 days. Analysis, 40 days after extraction.	8081A 8082	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL 10% sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, 40 days of the start of the extraction.
	Solid	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.
	Waste	50 g	---	N/A	8081A 8082	4 or 8 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, 40 days of the start of the extraction.

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2,3</sup>		RCRA (SW846) <sup>3,4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Petroleum Hydrocarbons /Oil and Grease	Water	1 L	1664A <sup>(7)</sup>	1 liter glass, Cool, 4°C HCl or H <sub>2</sub> SO <sub>4</sub> to pH <2 28 days	9071B	1 liter glass, Cool, 0-4°C HCl or H <sub>2</sub> SO <sub>4</sub> to pH <2 28 days
	Solid	30 g	1664A <sup>(7)</sup>	8 or 16 oz. Wide mouth glass jar, Cool, 4°C, 28 days	9071B	8 or 16 oz. wide mouth glass jar, Cool, 0-4°C, 28 days
	Waste	---	---	N/A	9071B	N/A
Purgeable Halocarbons by GC	Water	40 mL	601	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 14 days.	8021B	40 mL glass VOA vial (in triplicate) with Teflon®-lined septa with no headspace, Cool, 4°C, 1:1 HCl to pH ≤ 2, 14 days.
	Solid	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol). Cool, 4°C <sup>12</sup> .

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2, 3</sup>		RCRA (SW846) <sup>3, 4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Purgeable Halocarbons by GC (cont'd)	Waste	5 g or 25 g	---	N/A	8021B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Maximum holding time for Encore™ sampler is 48 hours (before the sample is added to methanol). Cool, 4°C <sup>12</sup>
Semivolatiles	Water	1L	625	1 liter amber glass with Teflon®-lined lid. Cool, 4°C. Extraction, 7 days. Analysis, 40 days.	8270C	1 liter amber glass with Teflon®-lined lid, If residual chlorine present, add 3 mL sodium thiosulfate per gallon. Cool, 4°C. Extraction, 7 days. Analysis, within 40 days of extraction.
	Solid	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.
	Waste	50 g	---	N/A	8270C	8 or 16 oz glass wide mouth with Teflon®-lined lid. Cool, 4°C. Extraction, 14 days. Analysis, within 40 days of extraction.

Analytical Parameters	Matrix	Minimum Sample Size <sup>1</sup>	NPDES <sup>2,3</sup>		RCRA (SW846) <sup>3,4</sup>	
			Method	Requirements	Method <sup>6</sup>	Requirements
Volatile Organics	Water	40 mL	624	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 7 days with pH > 2, 14 days with pH ≤ 2 <sup>8</sup> .	8260B	40 mL glass, VOA vial (in triplicate) with Teflon®-lined septa without headspace. Cool to 4°C. Add sodium thiosulfate if residual chlorine, 1:1 HCl to pH ≤ 2, 14 days with pH ≤ 2 <sup>9</sup> .
	Solid <sup>5</sup>	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid. Cool to 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for EnCore™ sampler is 48 hrs (before the sample is added to methanol or sodium bisulfate). Cool to 4°C <sup>(12)</sup>
	Waste	5 g or 25 g	--	N/A	8260B	4 or 8 oz glass with Teflon®-lined lid, Cool 4 °C, 14 days. Field preserved with sodium bisulfate solution for low level analysis, or with methanol for medium level analysis. Soil sample can also be taken by using the EnCore™ sampler and preserved in the lab within 48 hrs of sampling. Maximum holding time for Encore™ sampler is 48 hrs (before sample is added to methanol or sodium bisulfate). Cool to 4°C <sup>12</sup>

## Footnotes

---

- <sup>1</sup> Minimum sample size indicates sample amount needed for a single analysis. Matrix spikes or duplicates will require an additional sample amount of at least this amount for each additional QC sample aliquot required.
- <sup>2</sup> National Pollutant Discharge Elimination System - 40 CFR Part 136, Appendix A.
- <sup>3</sup> Holding times are calculated from the date of collection.
- <sup>4</sup> Resource Conservation and Recovery Act, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, September 1986. Contains Final Update I (July 1992), Final Update IIA (August 1993), Final Update II (September 1994), Final Update IIB (January 1995), and Final Update III (December 1996).
- <sup>5</sup> Solid matrix type includes soil, sediment, sludge or other solids not classified as waste.
- <sup>6</sup> Only one determination method is listed when separate methods are required for preparation and analysis.
- <sup>7</sup> Method 1664 was promulgated by the EPA with an effective date of June 14, 1999.
- <sup>8</sup> For acrolein and acrylonitrile the pH should be adjusted to 4-5. This pH adjustment is not required if acrolein is not measured. Samples requiring analysis of acrolein that received no pH adjustment must be analyzed within three days of sampling.
- <sup>9</sup> For acrolein and acrylonitrile the pH should be adjusted to 4-5.
- <sup>10</sup> Method not listed in 40 CFR Part 136.
- <sup>11</sup> Should only be used in the presence of residual chlorine.
- <sup>12</sup> Depending on regulatory programs, EnCore™ samplers may be preserved for up to 14 days from sampling by freezing at -5 to -12°C until analysis. Alternatively the EnCore™ sample may be transferred to a 40-ml VOA vial and preserved by freezing at -5 to -12°C until analysis. Some regulatory agencies may require 4 or 8 oz glass with Teflon®-lined lid, Cool 4°C, 14 days. This technique is not recommended, but will be supported where required. (Preservation and holding times are subject to client specifications.)

**Table 23-3.**

**Sample Containers, Preservatives, and Holding Times for TCLP<sup>1</sup> and SPLP<sup>2</sup>**

Analytical Parameters	Matrix	Minimum Sample Size	TCLP Method 1311 and SPLP Method 1312 Requirements	
			From Field Collection to TCLP/SPLP Extraction	From TCLP/SPLP Extraction to Analysis
Mercury	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 28 days	Glass or polyethylene 28 days
Metals (except mercury)	Liquid Solid Waste	1L	1L glass, Cool, 4°C, 180 days	Glass or polyethylene 180 days
Semivolatiles	Liquid Solid Waste	1L	1L glass, Cool 4°C, 14 days	1L glass Extraction of leachate within 7 days of TCLP extraction, Analyze extract within 40 days
Volatiles	Liquid Solid Waste	6 oz	4 oz glass, Cool 4°C, 14 days	40 mL glass, 14 days

**Footnotes**

<sup>1</sup> TCLP = Toxicity Characteristic Leaching Procedure

<sup>2</sup> SPLP = Synthetic Precipitation Leaching Procedure

## SECTION 24

### HANDLING OF SAMPLES (NELAC 5.5.8)

Sample management procedures at TestAmerica North Canton 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 can be 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.

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 COC is stored with project information and the report.

#### **24.1.2 Legal / Evidentiary Chain-of-Custody**

The lab does not accept samples that require legal chain-of-custody.

### **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. SOP NC-SC-0005, Sample Receiving and Sample Control, describes the laboratory's sample receipt procedure.

#### **24.2.1 Laboratory Receipt**

Samples shall be received and logged in at TestAmerica by a designated sample custodian or other properly trained associate. Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of cut-resistant gloves, a lab coat, and safety glasses
- Examine the shipping containers to verify that the custody tape is intact
- Examine all sample containers for damage
- Open shipping containers in adequately ventilated areas to assure worker safety
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the Cooler Receipt Form
- Compare samples received against those listed on the COC
- Verify that sample holding times have not been exceeded
- Examine all shipping records for accuracy and completeness
- Determine sample pH (if required for the scheduled analysis) (except VOA and TOX samples) and record on the Cooler Receipt Form (CRF)
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill
- Note any problems associated with the coolers and samples on the cooler receipt form and notify the PM who in turn notifies the client

- Attach durable (water-resistant) laboratory sample container labels with unique laboratory identification number and test
- Place the samples in proper laboratory storage.

A Cooler Receipt Form (CRF) or an equivalent form/system is generated by sample control during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The affected client shall be notified by the PM or designee of all CRFs generated for their samples. The PM is responsible for resolving with the client how to proceed with the samples and documenting the decision to proceed with the analysis of compromised samples. CRFs must be resolved prior to sample preparation and analysis. The completed CRF shall be stored in the project file. An example CRF is shown in Figure 24-4. The report narrative will include an explanation of sample receiving related anomalies.

#### **24.2.2 Exceptions or Discrepancies**

TestAmerica reserves the right to reject samples for any of the following reasons:

- No custody seals as required by project
- No chain of custody documentation provided
- Preservation inappropriate for analysis requested
- Sample container inappropriate for analysis requested
- Sample received out of holding time for analysis requested
- Incomplete sample information provided
- Discrepancies between COC and sample labels
- Samples have high levels of polychlorinated dibenzo-p-dioxins/ dibenzo furans (PCDD/PCDFs)
- Samples have a high level gross alpha or beta radiation
- Samples are from a site known to contain chemical warfare agents (CWAs) and the samples have not been screened for them.
- Samples containing high levels of PCBs, Cyanides, Sulfides, and Hydrofluoric Acid.
- Tissue samples that may contain viruses harmful to humans.
- Samples with percent levels of Target analytes must be discussed in advance before receipt.

These or any other project exceptions or discrepancies are discussed with the client and agreed upon action taken.

#### **24.2.3 Sample Log-In**

Sample log-in activities at TestAmerica North Canton are fully documented in SOP NC-SC-0005, Sample Receiving and Sample Control. The following is a general description of the log-in process:

- Client Name, Project Name, Address, Phone, Fax, Report to information, invoice to information. (Most of this information is “default information” that is stored in the LIMS.)
- Date and time sampled
- Date and time received
- Job and/or project description, sample description
- Sample matrix, special sample remarks
- Reporting requirements, i.e., QC level, report format, invoicing format
- Turn-around-time requirements
- Parameters (methods and reporting limits or MDLs are default information for a given parameter)

### **24.3 SAMPLE ACCEPTANCE POLICY**

The laboratory has a written sample acceptance policy outlined in SOP NC-SC-0005, Sample Receiving and Sample Control, 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.

### **24.4 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. Metals samples may be unrefrigerated. 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.

The primary considerations for sample storage are:

- Maintenance at the method prescribed temperature, if required
- Maintenance of sample integrity through adequate protection from contamination from outside sources or from cross-contamination of samples. Low-level and high-level samples,

when known, must be stored separately. Samples and standards must be stored in separate refrigerators or freezers. Storage areas for volatile organic test requests must be monitored weekly by the analysis of a holding (refrigerator) blank (an aliquot of contaminant-free water stored in a VOA vial)

- Security of samples within the laboratory.

The requirements listed in Tables 23-1 through 23-3 for temperatures and holding times shall be used. Placing of samples in the proper storage environment is the responsibility of sample control personnel. TestAmerica will assign individuals the responsibility of notifying the Group/Team Leaders or their designees if there are any samples which must be analyzed immediately because of holding time requirements.

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.

#### **24.5 HAZARDOUS SAMPLES AND FOREIGN SOILS**

All samples per SOP are treated as hazardous. If any extra or known hazards are present in the sample, the sample is flagged and precautions / instructions are put in the comments. Hazardous samples are segregated out, and go into the waste stream profile for the nature of the hazard. All soils--foreign and domestic--go to a USDA approved incinerator.

#### **24.6 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). 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.

#### **24.7 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 NC-SC-0005, Sample Receiving and Sample Control, and the Facility Addendum to the Corporate

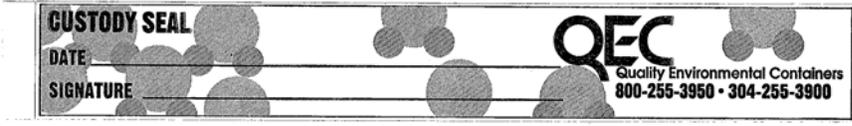
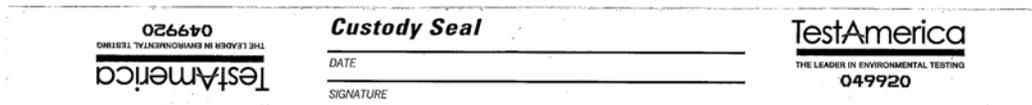
Safety Manual.) All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than two months from receipt unless otherwise requested. Unused portions of samples found or suspected to be hazardous according 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 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), and names of individuals who conducted the arrangements and physically completed the task. Sample labels are destroyed through the disposal method, e.g., samples are incinerated. A Waste Manifest is completed.



Figure 24-2.

Example: Custody Seals



**Figure 24-3.**

**Example: Internal Chain of Custody (COC)**

**TestAmerica Laboratories, Inc.  
Sample Control Record**

**Client:**

**Lot Number:**

**Case Number/SDG:**

**Storage Location:**

Laboratory Sample ID	Transferred By	Date	Entered	Removed	Reason	Date Returned

**Figure 24-4.**

**Example: Cooler Receipt Form (Page 1)**

TestAmerica Cooler Receipt Form/Narrative		Lot Number: _____	
North Canton Facility			
Client _____		Project _____ Quote # _____	
Cooler Received on _____		Opened on _____ By _____	
FedEx <input type="checkbox"/> Client Drop Off <input type="checkbox"/> UPS <input type="checkbox"/>		DHL <input type="checkbox"/> FAS <input type="checkbox"/> TestAmerica Courier <input type="checkbox"/>	
Stetson <input type="checkbox"/> US Cargo <input type="checkbox"/>		Other _____ (Signature)	
TestAmerica Cooler # _____		Foam Box <input type="checkbox"/> Client Cooler <input type="checkbox"/> Other _____	
1. Were custody seals on the outside of the cooler? Yes <input type="checkbox"/> No <input type="checkbox"/>		Intact? Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
If YES, Quantity _____			
Were custody seals on the outside of cooler signed and dated?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
Were custody seals on the bottles?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
If YES, are there any exceptions _____			
2. Shipper's packing slip attached to this form?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
3. Did custody papers accompany the sample(s)? Yes <input type="checkbox"/> No <input type="checkbox"/>		Relinquished by client? Yes <input type="checkbox"/> No <input type="checkbox"/>	
4. Did you sign the custody papers in the appropriate place?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
5. Packing material used: Bubble Wrap <input type="checkbox"/> Foam <input type="checkbox"/> None <input type="checkbox"/>		Other _____	
6. Cooler temperature upon receipt _____ °C (see back of form for multiple coolers/temps)			
METHOD: IR <input type="checkbox"/> Other <input type="checkbox"/>			
COOLANT: Wet Ice <input type="checkbox"/> Blue Ice <input type="checkbox"/> Dry Ice <input type="checkbox"/> Water <input type="checkbox"/> None <input type="checkbox"/>			
7. Did all bottles arrive in good condition (Unbroken)?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
8. Could all bottle labels and/or tags be reconciled with the COC?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
9. Were samples at the correct pH upon receipt?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
10. Were correct bottles used for the tests indicated?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
11. Were air bubbles >6 mm in any VOA vials?		Yes <input type="checkbox"/> No <input type="checkbox"/> NA <input type="checkbox"/>	
12. Sufficient quantity received to perform indicated analyses?		Yes <input type="checkbox"/> No <input type="checkbox"/>	
13. Was a Trip Blank present in the cooler? Yes <input type="checkbox"/> No <input type="checkbox"/>		Were VOAs on the COC? Yes <input type="checkbox"/> No <input type="checkbox"/>	
Contacted PM _____ Date _____ by _____ via Voice Mail <input type="checkbox"/> Verbal <input type="checkbox"/> Other <input type="checkbox"/>			
Concerning _____			
<b>14. CHAIN OF CUSTODY</b>			
The following discrepancies occurred: _____ _____ _____			
<b>15. SAMPLE CONDITION</b>			
Sample(s) _____ were received after the recommended holding time had expired.			
Sample(s) _____ were received in a broken container.			
<b>16. SAMPLE PRESERVATION</b>			
Sample(s) _____ were further preserved in sample receiving to meet recommended pH level(s). Nitric Acid Lot #071707-HNO3 - Sulfuric Acid Lot # 092006-H2SO4; Sodium Hydroxide Lot # 122805 -NaOH; Hydrochloric Acid Lot # 092006-HCl; Sodium Hydroxide and Zinc Acetate Lot # 050205-CH3COO2ZN/NaOH			
What time was preservative added to sample(s)? _____			
Sample(s) _____ were received with bubble > 6 mm in diameter (Notify PM)			
<b>Client ID</b>	<b>pH</b>	<b>Date</b>	<b>Initials</b>



## SECTION 25.0

### 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 21, 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 may 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.

#### 25.3 NEGATIVE CONTROLS

**25.3.1 Method Blanks** are used to assess preparation and analysis for possible contamination during the preparation and processing steps.

**25.3.1.1** 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.

**25.3.1.2** The method blank goes through all of the steps of the process, including as necessary, filtration, clean-ups, etc.

**25.3.1.3** 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 one for each batch of samples--not to exceed 20 environmental samples.

**25.3.1.4** Evaluation criteria and corrective action for method blanks is defined in the specific standard operating procedure for each analysis. Generally, corrective action is taken if the concentration of a target analyte in the blank is at or above the reporting limit as established by the method or regulation. Refer to Policy QA-003, TestAmerica North Canton Quality Control Program.

- The source of contamination is investigated
- Measures are taken to minimize or eliminate the source of the contamination
- Affected samples are reprocessed or the results are qualified on the final report.

**25.3.2** 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.

**25.3.3** 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.

**25.3.4** Trip Blanks are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses. 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. Trip Blanks are also sometimes referred to as Travel Blanks.

**25.3.5** Field Blanks 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)

**25.3.6** Equipment Blanks 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)

**25.3.7** 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. Refer to SOP NC-QA-0020, Laboratory Holding Blanks, for additional information on holding blank criteria.

**25.3.8** Field blanks, equipment blank and trip blanks, when received, are analyzed in the same manner as other field samples. When known, blanks 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".

## **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
- 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 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** As stated in the opening of this section, the LCS goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).
- 25.4.1.5** 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 one for each batch of samples; not to exceed 20 environmental samples.
- 25.4.1.6** 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 (see below) shall 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.

**25.4.1.6.1** For methods that have 1-10 target analytes, spike all components.

**25.4.1.6.2** For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.

**25.4.1.6.3** For methods with more than 20 target analytes, spike at least 16 components.

**25.4.1.6.4** Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.

**25.4.1.6.5** 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.

**25.4.1.7** **Accuracy Calculation**: Percent Recovery (%R) Calculation (applies to LCS, CCV, Surrogates, and Matrix Spikes).

$$\%R = \frac{AV}{TV} \times 100$$

Where: AV = Analyzed Value  
TV = True Value

## **25.5** **SAMPLE MATRIX CONTROLS**

### **25.5.1** **Matrix Spikes (MS)**

**25.5.1.1** The Matrix spike is 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.

**25.5.1.2** An MS is essentially a sample fortified with a known amount of the test analyte(s). 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.

**25.5.1.3** 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 of the listed

components (see LCS analytes in Section 25.4.1.6 above) 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.

- 25.5.1.4** The percent recovery calculation for matrix spikes is essentially the same as the calculation shown in Section 25.4.1.7, except that:

$$AV = Sp - Sa$$

Where: Sp = Spike result  
Sa = Sample result

### **25.5.2** Surrogate Spikes

- 25.5.2.1** Surrogate Spikes 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.

- 25.5.2.2** Surrogate compounds 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 (also refer to Section 25.5). 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.

### **25.5.3** Duplicates

- 25.5.3.1** 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. Duplicate samples are usually analyzed with methods that do not require matrix spike analysis. 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.

- 25.5.3.2** Precision Calculation (Relative Percent Difference - RPD)

$$RPD = \frac{|S - D|}{\frac{(S + D)}{2}} \times 100$$

Where: S=Sample Concentration  
D=Duplicate Concentration

#### **25.5.4 Internal Standards**

**25.5.4.1** In most organic analyses, internal standards are spiked into all environmental and quality control samples (including the initial calibration standards). An internal standard is also used with some metals analyses. It is added to sample extracts after the extraction (post-prep). The acceptance criteria in most methods are 50% to 200% of the responses in the mid-point of the corresponding calibration curve. Consult the method-specific SOPs for details on the internal standard compounds, calculations and acceptance criteria.

**25.5.4.2** When the internal standard recoveries fall outside these limits, if there are not obvious chromatographic interferences, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets internal standard recovery criteria, the second run is reported (or both are reported if requested by the client).

#### **25.6 ACCEPTANCE CRITERIA (CONTROL LIMITS)**

**25.6.1** Each individual analyte in the LCS, MS, or Surrogate Spike are evaluated against the control limits as published in the test method. Where there are no established acceptance criteria, the laboratory calculates control limits with the use of control charts or, in some cases, utilizes client project specific or regulatory mandated 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.

**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 (e.g. EPA SW846 8000 series methods). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

**25.6.2.1** The lab should consider the effects of the spiking concentration control limits, and to avoid censoring of data. The acceptance criteria for recovery and precision are often a function of the spike concentration used. Therefore, caution must be used when pooling data to generate control limits.

**25.6.2.2** Not only should the results all be from a similar matrix, but the spiking levels should also be approximately the same (within a factor of 2). Similarly, the matrix spike and surrogate results should all be generated using the same set of extraction, cleanup and analysis techniques. For example, results from solid samples extracted by ultrasonic extraction are not mixed with those extracted by Soxhlet.

**25.6.2.3** The laboratory should try and avoid discarding data that do not meet a preconceived notion of acceptable performance. This results in a censored data set, which, when used to develop acceptance criteria, will lead to unrealistically narrow criteria. For a 99% confidence interval, one out of every 100 observations likely will still fall outside

the limits. For methods with long analyte lists this may mean occasional failures every batch or two. While professional judgment is important in evaluating data to be used to develop acceptance criteria, specific results are not discarded simply because they do not meet one's expectations. However, data points shall be discarded if they were the result of human or mechanical error or sample concentration exceeded spike level by  $> 4x$ .

**25.6.3** 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).

**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.

**25.6.3.3** The lowest acceptable recovery limit will be 10% (the analyte must be detectable).

**25.6.3.4** The maximum acceptable recovery limit will be 200%.

**25.6.3.5** The maximum acceptable RPD limit will be 30% for organic methods and 20% for inorganic methods. The minimum RPD limit is 10%.

**25.6.3.6** If either the high or low end of the control limit changes by  $\leq 10\%$  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. Refer to NC-QA-0018, Statistical Evaluation of Data and Development of Control Charts, for details.

**25.6.4.1** One example: The QA Department generates a Quality Control Limit Summary that contains tables that summarize the precision and accuracy acceptability limits for analyses. Unless otherwise noted, limits within these tables are laboratory generated. Once reviewed and approved, the limits are entered into LIMS and are effective immediately. 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.
- 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 exceedances 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 Appendix 4 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 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).

## **25.7        METHOD DETECTION LIMITS (MDLs)**

MDLs, calculated as described in Section 20.7, are updated or verified annually, or more often if required by the method.

## **25.8        ADDITIONAL PROCEDURES TO ASSURE QUALITY CONTROL**

**25.8.1** The laboratory has written procedures 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).

**25.8.2** A discussion regarding MDLs, Limit of Detection (LOD), and Limit of Quantitation (LOQ) can be found in Section 20.

**25.8.3** Use of formulae to reduce data is discussed in the method Standard Operating Procedures and in Section 21.

**25.8.4** Selection of appropriate reagents and standards is included in Sections 9 and 22.

**25.8.5** A discussion on selectivity of the test is included in Section 5.

**25.8.6** Constant and consistent test conditions are discussed in Section 19.

**25.8.7** The laboratory sample acceptance policy is included in Section 24.

## **SECTION 26.0**

### **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 a conflict between the client requested formats and accreditation requirements or data usability information, accreditation requirements and data usability information will take precedence over client requests. 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 TEST REPORTS**

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 Project Manager. At a minimum, the standard laboratory report shall contain the following information:

**26.2.1** A report title with a "Sample Result" header.

**26.2.2** Each report page printed 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 at the bottom of each page. The report is sequentially paginated. The final page of the report is labeled as "End of Report".

**26.2.4** 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 (e.g. Sampling information).

**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)

**26.2.11** 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 Section 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 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.

**26.2.19** 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.

**26.2.20** The laboratory includes a cover page.

**26.2.21** 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.

**26.2.22** When Soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**26.2.23** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**26.2.24** 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 will follow once all of the work has been completed.

**26.2.25** Any out-of-network subcontracted analysis results are provided as a separate report on the official letterhead of the subcontractor. All in-network subcontracting is clearly identified on the report as to which laboratory performed a specific analysis.

### **26.3 REPORTING LEVEL OR REPORT TYPE**

TestAmerica North Canton offers two 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:

- Standard report – all features in Section 26.2
- Expanded deliverable – standard report

Presented on CLP-like forms and relevant calibration information. All supporting raw data is supplied.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Procedures used to ensure client confidentiality are outlined in Section 26.7.

#### **26.3.1 Electronic Data Deliverables (EDDs)**

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica North Canton offers a variety of EDD formats.

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.

## **26.4 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. Refer to Appendix 8 for a list of the laboratory's standard footnotes and qualifiers.

**26.4.1** Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

**26.4.2** Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications, 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.

**26.4.3** Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

**26.4.4** 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.

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.

## **26.5 ENVIRONMENTAL TESTING OBTAINED FROM SUBCONTRACTORS**

If TestAmerica North Canton 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 the TestAmerica network are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

## **26.6 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.

**26.6.1** 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:

"Confidentiality Notice: The information contained in this message is intended only for the use of the addressee, and may be confidential and/or privileged. If the reader of this message is not the intended recipient, or the employee or agent responsible to deliver it 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 immediately."

## **26.7**      **FORMAT OF REPORTS**

The format of reports are designed to accommodate each type of environmental test carried out and to minimize the possibility of misunderstanding or misuse.

## **26.8**      **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 13).

If, after issuance of a report, TestAmerica North Canton observes any mistake that affects the results reported or the QC interpretation of those results, the client will be notified. After issuance of the report, the laboratory report remains unchanged. Any material amendments to a report after issue made only in the form of a further document, or data transfer must include the statement "Supplement to Test Report" or otherwise identified.

When the report is re-issued, a notation 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.

## **26.9**      **POLICIES ON CLIENT REQUESTS FOR AMENDMENTS**

### **26.9.1**   **Sample Reanalysis Policy**

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 arrangements for reanalysis protocols can be established.

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within  $\pm 1$  reporting limit for samples  $\leq 5x$  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 Non-homogenous, Encore, and Sodium Bisulfate preserved samples. See the Group Leader, QA Manager, or Laboratory Director/Manager if unsure.

#### **26.9.2 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 no possible impact on the interpretation of the analytical results and there is no possibility of the change being interpreted as misrepresentation by anyone inside or outside of our company.

#### **26.9.3 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.

**Appendix 1. TestAmerica Ethics Policy CA-L-P-001**

**TESTAMERICA  
ETHICS POLICY No. CA-L-P-001**

Refer to CA-L-P-001 for complete policy.

**TestAmerica  
EMPLOYEE ETHICS STATEMENT**

*I understand that TestAmerica is committed to ensuring the highest standard of quality and integrity of the data and services provided to our clients. I have read the Ethics Policy of the Company.*

- *With regard to the duties I perform and the data I report in connection with my employment at the Company, I agree that:*
- *I will not intentionally report data values that are inconsistent with the actual values observed or measured.*
- *I will not intentionally report the dates, times, sample or QC identifications, or method citations of data analyses that are not the actual dates, times, sample or QC identifications, or method citations.*
- *I will not intentionally misrepresent another individual's work as my own or represent my own work as someone else's.*
- *I will not intentionally misrepresent any data where data does not meet Method or QC requirements. If it is to be reported, I will report it with all appropriate notes and/or qualifiers; I shall not modify data (either sample or QC data) unless the modification can be technically justified through a measurable analytical process, such as one deemed acceptable to the laboratory's Standard Operating Procedures, Quality Assurance Manual or Technical Director. All such modifications must be clearly and thoroughly documented in the appropriate laboratory notebooks/worksheets and/or raw data and include my initials or signature and date.*
- *I shall not make false statements to, or seek to otherwise deceive, members of Management or their representatives, agents, or clients/customers. I will not, through acts of commission, omission, erasure, or destruction, improperly report measurement standards, quality control data, test results or conclusions.*
- *I shall not compare or disclose results for any Performance Testing (PT) sample, or other similar QA or QC requirements, with any employee of any other laboratory, including any other TestAmerica laboratory, prior to the required submission date of the results to the person, organization, or entity supplying the PT sample.*
- *I shall immediately inform my supervisor or other member of management regarding any intentional or unintentional reporting of my own inauthentic data. Such report shall be given both orally and in writing to the supervisor or other member of management contacted and to the local Quality Assurance Manager. The Quality Assurance Manager will initial and date the information and return a copy to me. I shall not condone any accidental or intentional reporting of inauthentic data by other employees and will immediately report its occurrence. If I have actual knowledge of such acts committed by any other employees, and I do not report such information to designated members of Management, it shall be considered as serious as if I personally committed the offense. Accordingly, in that event, I understand that I may be subject to immediate termination of employment.*

- *I understand that if any supervisor, manager, or representative of TestAmerica management instructs, requests, or directs me to perform any of the aforementioned improper laboratory practices, or if I am in doubt or uncertain as to whether or not such laboratory practices are proper, I will not comply. In fact, I must report such event to all appropriate members of Management including, but not limited to, the Lab Director, all supervisors and managers with direct line reporting relationship between me and the Lab Director, and the local Quality Assurance representative, excluding such individuals who participated in such perceived improper instruction, request, or directive. In addition, I may contact Corporate Quality Assurance / Ethics Compliance Officer(s) for assistance.*
- *I understand the critical importance of accurately reporting data, measurements, and results, whether initially requested by a client, or retained by TestAmerica and submitted to a client at a later date, or retained by TestAmerica for subsequent internal use;*
- *I will not share the pricing or cost data of Vendors or Suppliers with anyone outside of the TestAmerica family of companies.*
- *I shall not accept gifts of a value that would adversely influence judgment.*
- *I shall avoid conflicts of interest and report any potential conflicts to the management (e.g. employment or consulting with competitors, clients, or vendors).*
- *I shall not participate in unfair competition practices (e.g. slandering competitors, collusion with other labs to restrict others from bidding on projects).*
- *I shall not misrepresent certifications and status of certifications to clients or regulators.*
- *I shall not intentionally discharge wastes illegally down the drain or onto the ground.*
- *I understand that any attempt by management or an employee to circumvent these policies will be subject to disciplinary action.*

*As a TestAmerica employee, I understand that I have the responsibility to conduct myself with integrity in accordance with the ethical standards described in the Ethics Policy. I will also report any information relating to possible kickbacks or violations of the Procurement Integrity Act, or other questionable conduct in the course of sales or purchasing activities. I will not knowingly participate in any such activity and will report any actual or suspected violation of this policy to management.*

*I understand that if my job includes supervisory responsibilities, I shall not instruct, request, or direct any subordinate to perform any laboratory practice which is unethical or improper. Also, I shall not discourage, intimidate, or inhibit an employee who may choose to appropriately appeal my supervisory instruction, request, or directive which the employee perceives to be improper, nor retaliate against those who do.*

*The Ethics Policy has been explained to me by my supervisor or at a training session, and I have had the opportunity to ask questions if I did not understand any part of it. I understand that any violation of this policy subjects me to disciplinary action, which can include termination of my employment. In addition, I understand that any violation of this policy which relates to work under a government contract or subcontract could also subject me to the potential for prosecution under federal law.*

EMPLOYEE SIGNATURE \_\_\_\_\_

Date \_\_\_\_\_

Supervisor/Trainer: \_\_\_\_\_

Date \_\_\_\_\_

Work Instruction No. CA-WI-005

**TestAmerica**  
**CONFIDENTIALITY AND PROPRIETARY INFORMATION AGREEMENT**

TestAmerica and their predecessors, in their businesses, have developed and use commercially valuable technical and non-technical information and to guard the legitimate interests of TestAmerica and its clients, it is necessary to protect certain information as confidential and proprietary.

I, \_\_\_\_\_, understand and acknowledge that during the term of my employment by TestAmerica, I will be privy to and entrusted with certain confidential information and trade secrets of TestAmerica and its clients.

*Confidential information and trade secrets include, but are not limited to: customer and client lists; price lists; marketing and sales strategies and procedures; operational and equipment techniques; standard operating procedures; business plans and systems; quality control procedures and systems; special projects and technological research, including projects, research and reports for any government entity or client; client's plans and processes; client's manner of operation; the trade secrets of clients; client's data; vendor or supplier pricing; employee lists and personal information, and any other records, data, files, drawings, inventions, discoveries, applications, or processes which are not in the public domain.*

I agree as follows:

1. I will not in any way, during the term of my employment, or at any time thereafter, except as authorized in writing by the Legal Department of TestAmerica or the client where client data is involved, disclose to others, use for my own benefit, remove from TestAmerica's premises (except to the extent off-site work is approved by my supervisor), copy or make notes of any confidential information and/or trade secrets of TestAmerica or its clients, excepting only that information which may be public knowledge. Technical and business information of any previous employer or other third party which I may disclose to TestAmerica shall be limited to that which was acquired legitimately and disclosed to me without restriction as to secrecy.
2. I agree that all inventions (whether or not patentable) conceived or made by me during the period of my employment by TestAmerica shall belong to TestAmerica, provided such inventions grow out of my work for TestAmerica and are related to the business of TestAmerica. I agree to disclose and assign such inventions to TestAmerica. In California, this provision shall not apply to any invention which qualifies fully under Section 2870 of the California Labor Code.
3. On termination of my employment from TestAmerica, I will deliver to TestAmerica all documents, records, notes, data, memoranda, files, manuals, equipment and things of any nature which relate in any way to confidential information and/or trade secrets of TestAmerica or its clients and which are in my possession or under my control.
4. I agree that during the period of my employment and for one (1) year from and after the termination (for any reason) of my employment with TestAmerica, I shall not directly or indirectly (without first obtaining the written permission of TestAmerica), recruit for employment, or induce to terminate his or her employment with TestAmerica, any person who is an active employee of TestAmerica on the last day of my employment with TestAmerica.
5. I acknowledge that if I were to breach any provision of this Confidentiality Agreement, money damages will be inadequate, and I hereby agree that TestAmerica shall be entitled, where appropriate, to specific performance and/or injunctive relief (i.e. to require me to comply with this Agreement). I further acknowledge that the willingness of TestAmerica to hire me or to continue my employment constitutes full and adequate consideration for the agreements, and obligations to which I have agreed as set forth in this document.

I have executed this Agreement, intending to be legally bound.

\_\_\_\_\_  
Printed Name

\_\_\_\_\_  
Signature

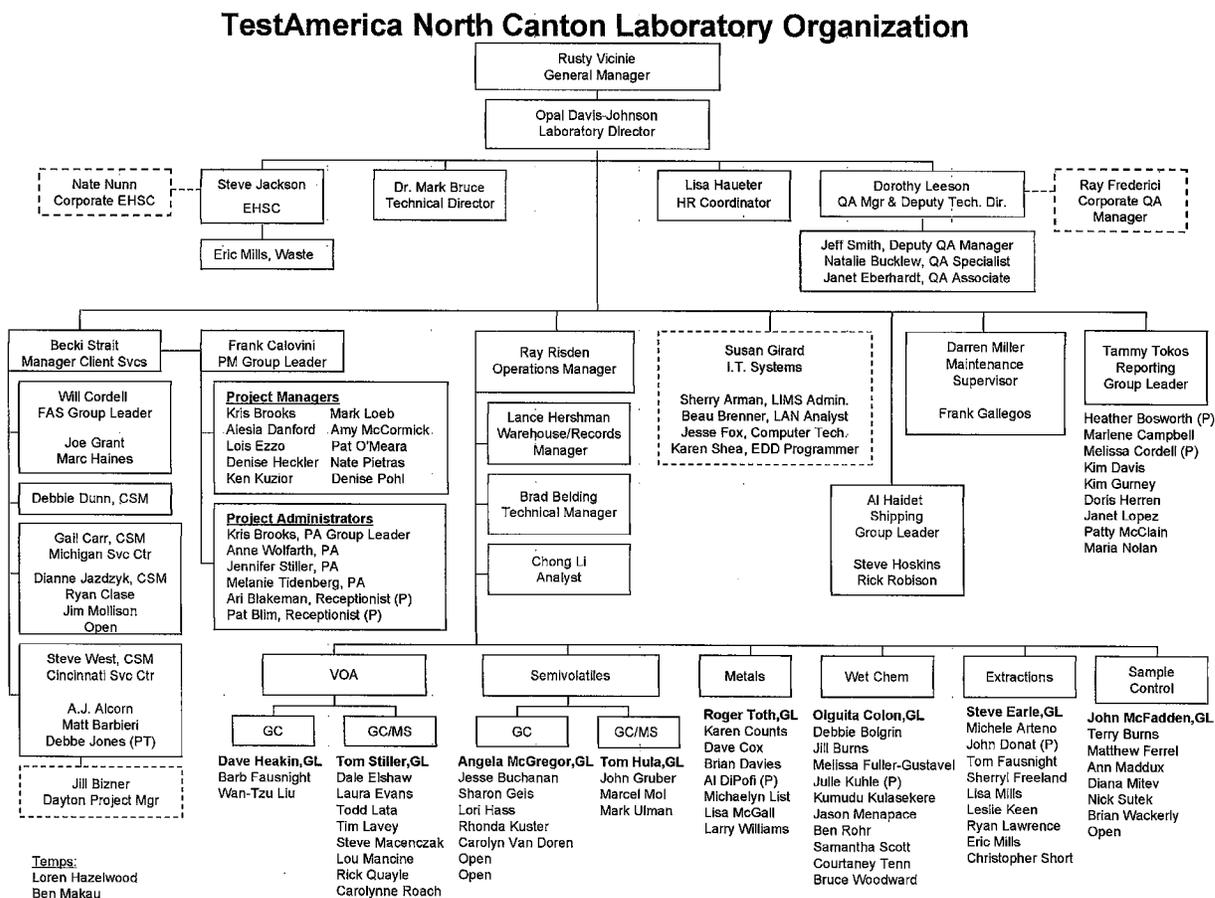
\_\_\_\_\_  
Date

Work Instruction No. CA-WI-006

**Appendix 2.**

**Example Laboratory Organization Chart**

(The most current chart can be obtained from the QA Manager or Lab Director/Manager)



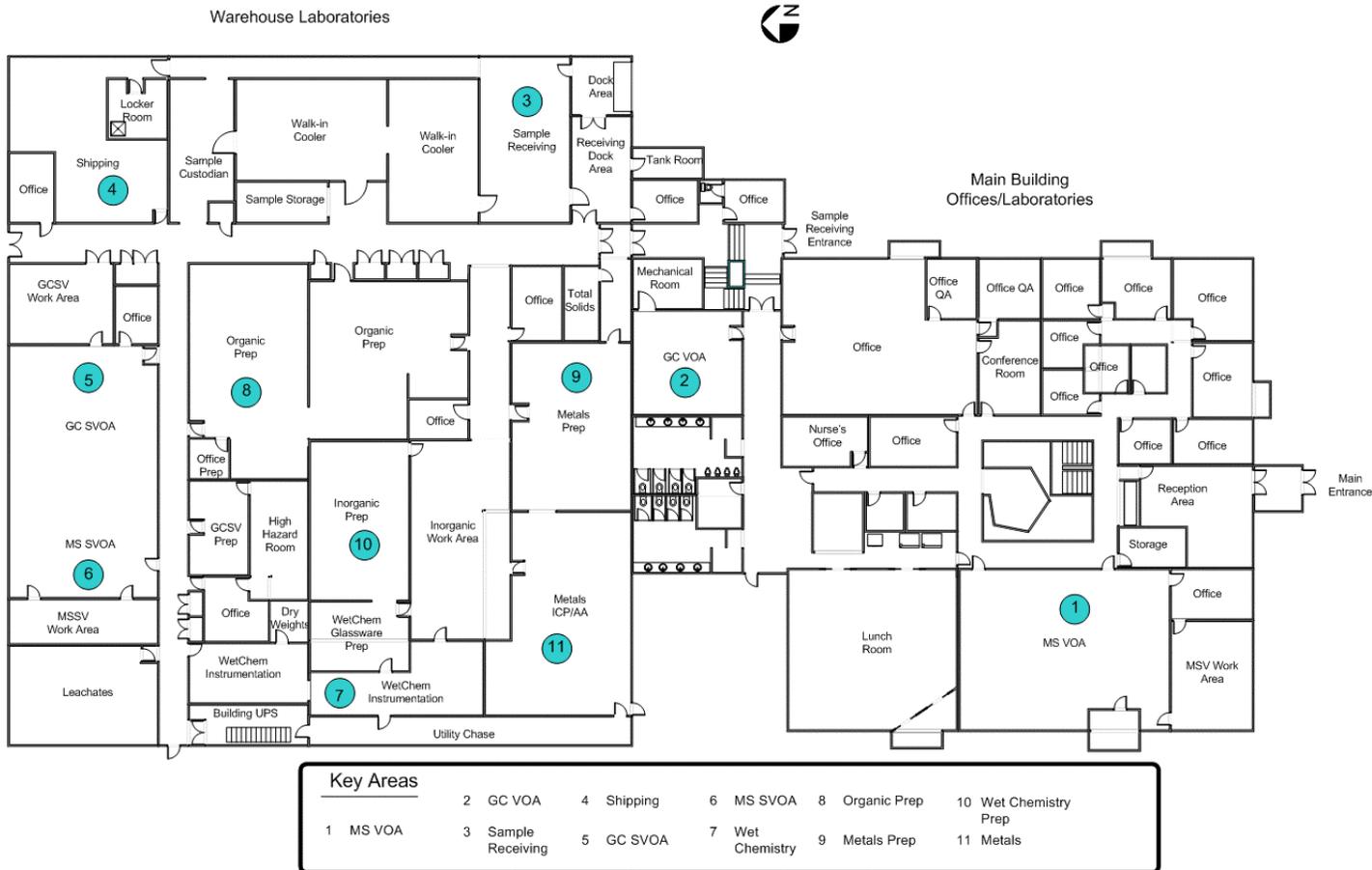
11/06/07

**Appendix 3.**

**Laboratory Floor Plan**

**TestAmerica – North Canton**

4101 Shuffel Dr NW  
 North Canton, OH 44720



**Appendix 4.**

**Laboratory Method Listing**

**Wet Chemistry Methods <sup>1</sup>**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Acidity	Water	305. <sup>2</sup> SM 2310 B	--	--
Alkalinity, Bicarbonate, Carbonate	Water	305. <sup>2</sup> SM 2320 B		
	Solid	EPA 310.1 <sup>2</sup> (M)	--	--
Arsenic (ASV) Anodic Stripping Voltammetry	Water	--	EPA 7063	--
Ash Content	Solid	--	--	ASTM D29-74
Biochemical Oxygen Demand, Carbonaceous	Water	EPA 405.1 SM 5210 B	--	--
Bromide	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Cation-Exchange Capacity	Solid	--	EPA 9081	--
Chemical Oxygen Demand	Water	EPA 410.4 SM 5220D	--	--
	Waste	EPA 410.4	--	--
Chloride	Water	EPA 300.0A EPA 325.2 <sup>2</sup>	EPA 9056A EPA 9251	EPA 325.2 <sup>2</sup>
	Waste	EPA 300.0A	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9251(M)	--
Chromium, Hexavalent	Water	EPA 3500-Cr-D	EPA 7196A	--
	Waste	EPA 3500-Cr-D	EPA 7196A	--
	Solid	--	EPA 3060A EPA 7196A	--

<sup>1</sup> Any matrix not listed is not applicable for the associated method

<sup>2</sup> Removed from 40CFR

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Specific Conductance	Water	EPA 120.1 SM 2510B	EPA 9050A	--
	Waste	EPA 120.1	EPA 9050A	--
	Solid	--	EPA 9050A	--
Chlorine, Residual	Water	EPA 330.5 <sup>2</sup> SM 3500 CL-G	--	--
Cyanide (Amenable)	Water	EPA 335.1 <sup>2</sup> SM 4500 CN-G	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Total)	Water	SM 4500-CN E EPA 335.4	EPA 9012A	---
	Waste	--	EPA 9012A	--
	Solid	--	EPA 9012A	--
Cyanide (Weak and Dissociable) (Free)	Water	SM 4500-CN I	--	--
Dissolved Oxygen	Water	360.1 <sup>2</sup> SM 4500 O-G	--	--
Flash Point	Waste	--	EPA 1010	ASTM D93-9
	Solid	--	EPA 1010	ASTM D93-9
Fluoride	Water	EPA 300.0A EPA 340.2 <sup>2</sup>	EPA 9056A	SM 4500 F-C, ISE
	Waste	EPA 340.2 (M) <sup>2</sup> EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 340.2 (M) <sup>2</sup> EPA 300.0A (M)	EPA 9056A	--
Iron, Ferrous & Ferric	Water	SM 3500 FE D	--	--
Hardness	Water	EPA 130.2 <sup>2</sup>	--	SM 2340B
Moisture	Solid	---	EPA 160.3 (M) ASTM D2216-90	---
Nitrogen, Ammonia	Water	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Waste	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Solid	EPA 350.1	--	EPA 350.2 <sup>2</sup>
	Water	SM 4500 NH <sub>3</sub> -B (Distillation)	--	--
	Water	SM 4500 NH <sub>3</sub> -E (Titration)	--	--
	Water	SM 4500 NH <sub>3</sub> -F (ISE)	--	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Nitrite (NO <sub>2</sub> )	Water	EPA 300.0A	EPA 9056A	--
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	EPA 9056A	--
Nitrate (NO <sub>3</sub> )	Water	EPA 300.0A	EPA 9056A	SM 4500 NO <sub>3</sub> -E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M)	--	--
Nitrate plus Nitrite NO <sub>2</sub> /NO <sub>3</sub>	Water	EPA 353.2	--	--
	Waste	EPA 353.2	--	--
Total Kjeldahl Nitrogen (TKN)	Water	EPA 351.3	--	SM 4500 NO <sub>3</sub>
	Waste	EPA 351.3	--	--
	Solid	EPA 351.3	--	--
Oil and Grease (Hexane Extractable Material)	Water	EPA 1664A	EPA 9071B	--
	Waste	EPA 1664A	EPA 9071B	--
	Solid	--	EPA 9071B	--
Ortho-phosphate o-PO <sub>4</sub>	Water	EPA 300.0A EPA 365.1	EPA 9056A	SM 4500 P-E
	Waste	EPA 300.0A (M)	EPA 9056A	--
	Solid	EPA 300.0A (M) EPA 365.1	EPA 9056A	--
pH	Water	EPA 150.1 <sup>2</sup>	EPA 9040B	EPA 9041
	Waste	SM 4500 H-B	EPA 9045C	--
	Solid	---	EPA 9045C	--
Paint Filter	Water	--	EPA 9095A	--
Phenolics	Water	EPA 420.1	--	--
	Waste	--	EPA 9065	--
	Solid	--	EPA 9065	--
Phosphorus (Total)	Water	EPA 365.1	--	SM 4500 P-E
	Waste	EPA 365.1	--	--
	Solid	EPA 365.1	--	--
Sulfate (SO <sub>4</sub> )	Water	EPA 300.0A EPA 375.4 <sup>2</sup>	EPA 9056A EPA 9038	--
	Waste	EPA 300.0A (M) EPA 375.4 <sup>2</sup>	EPA 9056A EPA 9038	--
	Solid	EPA 300.0A (M)	EPA 9056A EPA 9038 (M)	--

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA	Other
Sulfide	Water	EPA 376.1 <sup>2</sup>	EPA 9030A SM 4500	9030B/9034
Total Organic Carbon (TOC)	Water	EPA 415.1 <sup>2</sup>	EPA 9060	SM 5310 D
	Waste	--	EPA 9060	--
	Solid	EPA 415.1 (M)	EPA 9060 (M)	Walkley-Black
Total Organic Halides (TOX)	Water	--	EPA 9020B EPA 9023(EOX)	EPA 450.1
	Waste	--	--	--
	Solid	--	EPA 9020B	--
Total Petroleum Hydrocarbons	Water	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Waste	EPA 1664A (SGT-HEM)	EPA 9071B	--
	Solid	--	EPA 9071B	--
Total Solids	Water	EPA 160.3	--	--
	Waste	EPA 160.3	--	--
	Solid	EPA 160.3 (M)	--	--
Total Dissolved Solids	Water	EPA 160.1	--	2540E
Total Suspended Solids	Water	EPA 160.2	---	2540E
Volatile and Volatile Suspended Solids	Water	EPA 160.4	--	--
Settleable Solids	Water	EPA 160.5	--	--
Turbidity	Water	EPA 180.1	--	--

**Methods for Mercury by Cold Vapor Atomic Absorption**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury (CVAA)	Water	EPA 245.1	EPA 7470A	--
	TCLP Leachate	--	EPA 7470A	--
	Waste	--	EPA 7471A	--
	Solid	EPA 254.5	EPA 7471A	--

**Methods for Mercury by Cold Vapor Atomic Fluorescence**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Mercury, Low Level (CVAFS)	Water	EPA 245.7	--	EPA 1631E

**Methods for Metals by ICP and ICPMS**

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Aluminum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Antimony	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Arsenic	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Barium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Beryllium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Boron	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---
Calcium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	EPA 200.7	EPA 6010B	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Cadmium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Cobalt	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Chromium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Copper	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Iron	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Lead	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Magnesium	Water	EPA 200.7	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Manganese	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Molybdenum	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Nickel	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Potassium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Selenium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Silver	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Sodium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Tin	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Thallium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
Titanium	Water	EPA 200.7	EPA 6010B	---
	Waste	---	EPA 6010B	---
	Solid	---	EPA 6010B	---
Vanadium	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	---	EPA 6010B EPA 6020	---
Zinc	Water	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---
	Waste	---	EPA 6010B EPA 6020	---
	Solid	EPA 200.7 EPA 200.8	EPA 6010B EPA 6020	---

### Metals Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Toxicity Characteristic Leaching Procedure (TCLP)	Water	---	EPA 1311	---
	Waste	---	EPA 1311	---
	Solid	---	EPA 1311	---
ICP Metals	Water	EPA 200.7	EPA 3005A EPA 3010A	---
	TCLP Leachate	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
ICPMS Metals	Water	EPA 200.8	EPA 3010A	---
	TCLP	---	EPA 3010A	---
	Waste	---	EPA 3050B	---
	Solid	---	EPA 3050B	---
CVAA Mercury	Water	EPA 245.1	EPA 7470A	---
	TCLP Leachate	---	EPA 7470A	---
	Waste	---	EPA 7471A	---
	Solid	---	EPA 7471A	---
CVAFS Mercury Low Level	Water	EPA 245.7	---	EPA 1631E

### Organic Sample Preparation Methods

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 5030B	---
	Waste	---	EPA 5030B EPA 5035	---
	Solid	---	EPA 5035 EPA 5035A	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---
Pesticides/PCBs by GC	Water	EPA 608	EPA 3510C EPA 3520C	---
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3540C EPA 3580A EPA 3541	---
	Solid	---	EPA 3550B EPA 3540C EPA 3541	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Herbicides by GC	Water	EPA 615	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Total Petroleum Hydrocarbons (Gasoline Range) by GC	Water	---	EPA 5030B	WI GRO
	Waste	---	EPA 5030B EPA 5035	WI GRO
	Solid	---	EPA 5035 EPA 5035	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC	Water	---	EPA 3510C EPA 3520C	WI DRO
	TCLP Leachate	---	EPA 3510C EPA 3520C	---
	Waste	---	EPA 3550B EPA 3580A	WI DRO
	Solid	---	EPA 3550B	WI DRO

### Organic Methods of Analysis

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Volatiles by GC/MS	Water	EPA 624	EPA 8260B	---
	Waste	---	EPA 8260B	---
	Solid	---	EPA 8260B	---
Halogenated Volatiles by GC	Water	EPA 601	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Aromatic Volatiles by GC	Water	EPA 602	EPA 8021B	---
	Waste	---	EPA 8021B	---
	Solid	---	EPA 8021B	---
Semivolatiles by GC/MS	Water	EPA 625	EPA 8270C	---
	Waste	---	EPA 8270C	---
	Solid	---	EPA 8270C	---
Pesticides/PCBs by GC	Water	EPA 608	Pesticides 8081A PCBs 8082	---
	TCLP Leachate	---	Pesticides 8081A PCBs 8082	---
	Waste	---	Pesticides 8081A PCBs 8082	---
	Solid	---	Pesticides 8081A PCBs 8082	---

Analytical Parameters	Matrix	Fields of Testing		
		CWA	RCRA (SW846)	Other
Phenoxyacid Herbicides by GC	Water	---	EPA 8151A	---
	TCLP Leachate	---	EPA 8151A	---
	Waste	---	EPA 8151A	---
	Solid	---	EPA 8151A	---
Gasoline Range Organics by GC	Water	---	EPA 8015B (M)	WI GRO
	Waste	---	EPA 8015B (M)	---
	Solid	---	EPA 8015B (M)	WI GRO
Total Petroleum Hydrocarbons (Diesel Range) by GC/FID	Water	---	EPA 8015B (M)	WI DRO
	Waste	---	EPA 8015B (M)	---
Dissolved Gases RSK-175	Water	---	---	SOP
Formaldehyde Carbonyl Compounds	Water	---	EPA 8315	---

Appendix 5.

Laboratory Reporting Limits

**General Chemistry  
 Reporting Limits (RL), Method Reference**

Test Method	Analyte	RL	Units
305.1/SM2310B	Acidity	5	mg/L
310.1/SM2320B	Alkalinity, Total	5	mg/L
7063	Arsenic Speciation	2	ug/L
300.0 9056A	Bromide Chloride Nitrate Fluoride Sulfate Ortho-Phosphate Nitrite	0.5 1 0.1 1 1 0.5 0.1	mg/L mg/L mg/L mg/L mg/L mg/L mg/L
405.1/SM5210B	Biochemical Oxygen Demand	2	mg/L
410.4/SM5220D	Chemical Oxygen Demand (COD)	10	mg/L
325.2/9251	Chloride, Automated	1	mg/L
330.5 SM4500CL-G	Chlorine, Total Residue	0.2	mg/L
9012A/335.2 335.4 SM4500CN-E	Cyanide, Total	0.01	mg/L
340.2 SM4500F-C, ISE	Fluoride	0.1	mg/L
130.2/SM2340B	Hardness, as CaCO <sub>3</sub>	5	mg/L
1664A	n-Hexane Extractable Material n-Hexane Extractable Material, SGT	5 10	mg/L mg/L
7196A	Hexavalent Chromium	0.02	mg/L
3500 Cr D	Hexavalent Chromium	0.02	mg/L
353.2 SM4500NO <sub>3</sub> E	Nitrite Nitrate Nitrate/Nitrite	0.1 0.1 0.1	mg/L mg/L mg/L
350.2 SM4500NH <sub>3</sub> -E,F	Nitrogen, as Ammonia	1	mg/L

**General Chemistry  
 Reporting Limits (RL), Method Reference**

<b>Test Method</b>	<b>Analyte</b>	<b>RL</b>	<b>Units</b>
<b>9065</b>	Phenols, Total	0.02	mg/L
<b>9045B, C SM4500H-B</b>	pH (solid)	--	No Units
<b>4500</b>	Phosphorus as Orthophosphate	0.1	mg/L
<b>365.2 365.1</b>	Ortho-Phosphate Phosphorus, as ortho-Phosphate ortho-Phosphate, Dissolved	0.1 0.1 0.1	mg/L mg/L mg/L
<b>120.1 SM2510B</b>	Specific Conductance	1	umhos/cm
<b>9050A</b>	Specific Conductance	1	umhos/cm
<b>160.1 SM2540E</b>	Solids (Residue), Dissolved Filterable Solids, Total Dissolved	10 10	mg/L mg/L
<b>375.4</b>	Sulfate	5	mg/L
<b>9038</b>	Sulfate	5	mg/L
<b>376.1</b>	Sulfide, Total	1	mg/L
<b>9030A</b>	Sulfide, Total	1	mg/L
<b>9030A</b>	Sulfide, Acid-Insoluble	1	mg/L
<b>351.3</b>	Total Kjeldahl Nitrogen	3	mg/L
<b>415.1 SM5310D</b>	Total Organic Carbon	1	mg/L
<b>9060</b>	Total Organic Carbon	1	mg/L
<b>9020B</b>	Total Organic Halogens	30	ug/L
<b>180.1</b>	Turbidity	0.5	NTU

**Metals – ICP, CVAA 6000, 7000, 200 Series  
 Reporting Limits (RL), Method Reference<sup>1, 2</sup>**

	Analyte	RL	Units
<b>Trace Water</b>	Antimony	0.01	mg/L
	Arsenic	0.01	mg/L
	Cadmium	0.002	mg/L
	Chromium	0.005	mg/L
	Cobalt	0.007	mg/L
	Lead	0.003	mg/L
	Molybdenum	0.01	mg/L
	Selenium	0.005	mg/L
	Silver	0.005	mg/L
	Thallium	0.01	mg/L
	Vanadium	0.007	mg/L
<b>Trace Solid</b>	Antimony	1	mg/kg
	Arsenic	1	mg/kg
	Cadmium	0.2	mg/kg
	Chromium	0.5	mg/kg
	Cobalt	5	mg/kg
	Lead	0.3	mg/kg
	Molybdenum	1	mg/kg
	Selenium	0.5	mg/kg
	Silver	0.5	mg/kg
	Thallium	1	mg/kg
	Vanadium	5	mg/kg
<b>Water</b>	Aluminum	0.2	mg/L
	Antimony	0.06	mg/L
	Arsenic	0.3	mg/L
	Barium	0.2	mg/L
	Beryllium	0.005	mg/L
	Boron	0.2	mg/L
	Cadmium	0.005	mg/L
	Calcium	5	mg/L
	Chromium	0.01	mg/L
	Cobalt	0.05	mg/L
	Copper	0.025	mg/L
	Iron	0.1	mg/L
	Lead	0.1	mg/L
	Magnesium	5	mg/L
	Manganese	0.015	mg/L
	Molybdenum	0.04	mg/L
	Nickel	0.04	mg/L
	Potassium	5	mg/L
	Selenium	0.25	mg/L
	Silver	0.01	mg/L
	Sodium	5	mg/L
	Thallium	0.2	mg/L
Tin	0.1	mg/L	
Titanium	0.05	mg/L	
Vanadium	0.05	Mg/L	
Zinc	0.02	mg/L	

**TABLE 8.2-10-2**  
**Metals – ICP, CVAA 6000, 7000, 200 Series**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Solid</b>	Aluminum	20	mg/kg
	Antimony	6	mg/kg
	Arsenic	30	mg/kg
	Barium	20	mg/kg
	Beryllium	0.5	mg/kg
	Boron	20	mg/kg
	Cadmium	0.5	mg/kg
	Calcium	500	mg/kg
	Chromium	1	mg/kg
	Cobalt	5	mg/kg
	Copper	2.5	mg/kg
	Iron	10	mg/kg
	Lead	10	mg/kg
	Magnesium	500	mg/kg
	Manganese	1.5	mg/kg
	Molybdenum	4	mg/kg
	Nickel	4	mg/kg
	Potassium	500	mg/kg
	Selenium	25	mg/kg
	Silver	1	mg/kg
Sodium	500	mg/kg	
Thallium	200	mg/kg	
Tin	10	mg/kg	
Titanium	5	mg/kg	
Vanadium	5	mg/kg	
Zinc	2	mg/kg	
<b>Water</b>	Mercury	0.0002	mg/L
<b>Solid</b>	Mercury	0.1	mg/kg

**Metals – ICPMS, 6020**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum*	10	ug/L
	Nickel	2	ug/L
	Selenium*	5	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
Vanadium*	5	ug/L	
Zinc	10	Ug/L	

\*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

<b>Solid</b>	Antimony	0.2	mg/kg
	Arsenic	0.5	mg/kg
	Barium	0.1	mg/kg
	Beryllium	0.1	mg/kg
	Cadmium	0.1	mg/kg
	Chromium	0.2	mg/kg
	Cobalt	0.1	mg/kg
	Copper	0.2	mg/kg
	Lead	0.1	mg/kg
	Manganese	0.1	mg/kg
	Molybdenum*	0.2	mg/kg
	Nickel	0.1	mg/kg
	Selenium*	0.5	mg/kg
	Silver	0.1	mg/kg
	Thallium	0.1	mg/kg
	Tin*	1	mg/kg
	Vanadium*	0.5	mg/kg
	Zinc	1	mg/kg

\*Molybdenum, Selenium, Tin, and Vanadium are not on the 6020 method list

**Metals – ICPMS, 200.8  
 Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Aluminum	50	ug/L
	Antimony	2	ug/L
	Arsenic	5	ug/L
	Barium	1	ug/L
	Beryllium	1	ug/L
	Cadmium	1	ug/L
	Chromium	2	ug/L
	Cobalt	1	ug/L
	Copper	2	ug/L
	Iron	20	ug/L
	Lead	1	ug/L
	Manganese	1	ug/L
	Molybdenum	10	ug/L
	Nickel	2	ug/L
	Selenium	2	ug/L
	Silver	1	ug/L
	Thallium	1	ug/L
	Tin*	10	ug/L
	Vanadium	5	ug/L
Zinc	10	ug/L	

\*Tin is not on the 200.8 method list

**Metals – Low-Level Mercury, 1631E & 245.7  
 Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>1631E Water</b>	Mercury	0.5	ng/L
<b>245.7 Water</b>	Mercury	5	ng/L

**MS Semivolatiles – Method 8270C**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	Bis(2-chloroEthoxy)methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	Carbazole	10	ug/L
	4-Chloroaniline	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Dibenzofuran	10	ug/L
	Di-n-butyl phthalate	10	ug/L
<b>Water</b>	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	50	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	4,6-Dinitro-2-methylphenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloro-cyclopentadiene	50	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	2-Methylnaphthalene	10	ug/L
	2-Methylphenol	10	ug/L
	4-Methylphenol	10	ug/L
	Naphthalene	10	ug/L
2-Nitroaniline	50	ug/L	

	Analyte	RL	Units
	3-Nitroaniline	50	ug/L
	4-Nitroaniline	50	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodiphenylamine	10	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	2,2'-Oxybis(1-Chloropropane)	10	ug/L
	Pyrene	10	ug/L
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,5-Trichlorophenol	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
<hr/>			
<b>Water Surrogates</b>	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

**MS Semivolatiles – Method 8270C**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
	Acenaphthene	330	ug/kg
	Acenaphthylene	330	ug/kg
	Anthracene	330	ug/kg
	Benzo(a)anthracene	330	ug/kg
	Benzo(b)fluoranthene	330	ug/kg
	Benzo(k)fluoranthene	330	ug/kg
	Benzo(ghi)perylene	330	ug/kg
	Benzo(a)pyrene	330	ug/kg
	bis(2-Chloroethoxy)-methane	330	ug/kg
	bis(2-Chloroethyl) ether	330	ug/kg
	bis(2-Ethylhexyl) phthalate	330	ug/kg
	4-Bromophenyl phenyl ether	330	ug/kg
	Butyl benzyl phthalate	330	ug/kg
	Carbazole	330	ug/kg
	4-Chloroaniline	330	ug/kg
	4-Chloro-3-methylphenol	330	ug/kg
	2-Chloronaphthalene	330	ug/kg
	2-Chlorophenol	330	ug/kg
	4-Chlorophenyl phenyl ether	330	ug/kg
	Chrysene	330	ug/kg
<b>Solid</b>	Dibenz(a,h)anthracene	330	ug/kg
	Dibenzofuran	330	ug/kg
	Di-n-butyl phthalate	330	ug/kg
	1,2-Dichlorobenzene	330	ug/kg
	1,3-Dichlorobenzene	330	ug/kg
	1,4-Dichlorobenzene	330	ug/kg
	3,3'-Dichlorobenzidine	1600	ug/kg
	2,4-Dichlorophenol	330	ug/kg
	Diethyl phthalate	330	ug/kg
	2,4-Dimethylphenol	330	ug/kg
	Dimethyl phthalate	330	ug/kg
	4,6-Dinitro-2-methylphenol	1600	ug/kg
	2,4-Dinitrophenol	1600	ug/kg
	2,4-Dinitrotoluene	330	ug/kg
	2,6-Dinitrotoluene	330	ug/kg
	Di-n-octyl phthalate	330	ug/kg
	Fluoranthene	330	ug/kg
	Fluorene	330	ug/kg
	Hexachlorobenzene	330	ug/kg
	Hexachlorobutadiene	330	ug/kg
	Hexachloro-cyclopentadiene	1600	ug/kg
	Hexachloroethane	330	ug/kg
	Indeno(1,2,3-cd)pyrene	330	ug/kg
	Isophorone	330	ug/kg
	2-Methylnaphthalene	330	ug/kg
	2-Methylphenol	330	ug/kg
	4-Methylphenol	330	ug/kg
	Naphthalene	330	ug/kg
	2-Nitroaniline	1600	ug/kg
	3-Nitroaniline	1600	ug/kg

Analyte	RL	Units
4-Nitroaniline	1600	ug/kg
Nitrobenzene	330	ug/kg
2-Nitrophenol	330	ug/kg
4-Nitrophenol	1600	ug/kg
N-Nitrosodiphenylamine	330	ug/kg
N-Nitrosodi-n-propylamine	330	ug/kg
Pentachlorophenol	330	ug/kg
Phenanthrene	330	ug/kg
Phenol	330	ug/kg
2,2'-Oxybis(1-Chloropropane)	330	ug/kg
Pyrene	330	ug/kg
1,2,4-Trichlorobenzene	330	ug/kg
2,4,5-Trichlorophenol	330	ug/kg
2,4,6-Trichlorophenol	330	ug/kg

**Solid  
 Surrogates**

2-Fluorobiphenyl  
 2-Fluorophenol  
 2,4,6-Tribromophenol  
 Nitrobenzene-d5  
 Phenol-d5  
 Terphenyl-d14

**MS Semivolatiles – Method 625**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Acenaphthene	10	ug/L
	Acenaphthylene	10	ug/L
	Anthracene	10	ug/L
	Benzo(a)anthracene	10	ug/L
	Benzo(b)fluoranthene	10	ug/L
	Benzo(k)fluoranthene	10	ug/L
	Benzo(ghi)perylene	10	ug/L
	Benzo(a)pyrene	10	ug/L
	bis(2-Chloroethoxy)-methane	10	ug/L
	bis(2-Chloroethyl) ether	10	ug/L
	bis(2-Chloroisopropyl) ether	10	ug/L
	bis(2-Ethylhexyl) phthalate	10	ug/L
	4-Bromophenyl phenyl ether	10	ug/L
	Butyl benzyl phthalate	10	ug/L
	4-Chloro-3-methylphenol	10	ug/L
	2-Chloronaphthalene	10	ug/L
	2-Chlorophenol	10	ug/L
	4-Chlorophenyl phenyl ether	10	ug/L
	Chrysene	10	ug/L
	Dibenz(a,h)anthracene	10	ug/L
	Di-n-butyl phthalate	10	ug/L
	1,2-Dichlorobenzene	10	ug/L
	1,3-Dichlorobenzene	10	ug/L
	1,4-Dichlorobenzene	10	ug/L
	3,3'-Dichlorobenzidine	10	ug/L
	2,4-Dichlorophenol	10	ug/L
	Diethyl phthalate	10	ug/L
	2,4-Dimethylphenol	10	ug/L
	Dimethyl phthalate	10	ug/L
	2-Methyl-4,6-dinitrophenol	50	ug/L
	2,4-Dinitrophenol	50	ug/L
	2,4-Dinitrotoluene	10	ug/L
	2,6-Dinitrotoluene	10	ug/L
	Di-n-octyl phthalate	10	ug/L
	Fluoranthene	10	ug/L
	Fluorene	10	ug/L
	Hexachlorobenzene	10	ug/L
	Hexachlorobutadiene	10	ug/L
	Hexachloroethane	10	ug/L
	Indeno(1,2,3-cd)pyrene	10	ug/L
	Isophorone	10	ug/L
	Naphthalene	10	ug/L
	Nitrobenzene	10	ug/L
	2-Nitrophenol	10	ug/L
	4-Nitrophenol	50	ug/L
	N-Nitrosodi-n-propylamine	10	ug/L
	Pentachlorophenol	10	ug/L
	Phenanthrene	10	ug/L
	Phenol	10	ug/L
	Pyrene	10	ug/L

	Analyte	RL	Units
	1,2,4-Trichlorobenzene	10	ug/L
	2,4,6-Trichlorophenol	10	ug/L
<b>Water Surrogates</b>	2-Fluorobiphenyl		
	2-Fluorophenol		
	2,4,6-Tribromophenol		
	Nitrobenzene-d5		
	Phenol-d5		
	Terphenyl-d14		

**MS Volatiles – Method 8260B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water 25 mL</b>	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane	2	ug/L
	1,2-Dibromoethane	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoro-methane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/L
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	1	ug/L
	Naphthalene	1	ug/L
	n-Propylbenzene	1	ug/L
	Styrene	1	ug/L
	1,1,1,2-Tetrachloroethane	1	ug/L
	1,1,2,2-Tetrachloroethane	1	ug/L
	Tetrachloroethene	1	ug/L
	Toluene	1	ug/L
1,2,3-Trichlorobenzene	1	ug/L	
1,2,4-Trichlorobenzene	1	ug/L	
1,1,1-Trichloroethane	1	ug/L	
1,1,2-Trichloroethane	1	ug/L	

Analyte	RL	Units
Trichloroethene	1	ug/L
Trichlorofluoromethane	1	ug/L
1,2,3-Trichloropropane	1	ug/L
1,2,4-Trimethylbenzene	1	ug/L
1,3,5-Trimethylbenzene	1	ug/L
Vinyl chloride	1	ug/L
m-Xylene & p-Xylene	1	ug/L
o-Xylene	1	ug/L

---

<b>Water 25 mL Surrogates</b>	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

**MS Volatiles – Method 8260B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b> <b>5 mL</b>	Benzene	5	ug/L
	Bromobenzene	5	ug/L
	Bromochloromethane	5	ug/L
	Bromodichloromethane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	5	ug/L
	n-Butylbenzene	5	ug/L
	sec-Butylbenzene	5	ug/L
	tert-Butylbenzene	5	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Chlorodibromomethane	5	ug/L
	Chloroethane	5	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	2-Chlorotoluene	5	ug/L
	4-Chlorotoluene	5	ug/L
	1,2-Dibromo-3-chloropropane	10	ug/L
	1,2-Dibromoethane	5	ug/L
	Dibromomethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	Dichlorodifluoro-methane	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	cis-1,2-Dichloroethene	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	1,3-Dichloropropane	5	ug/L
	2,2-Dichloropropane	5	ug/L
	1,1-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Hexachlorobutadiene	5	ug/L
	Isopropylbenzene	5	ug/L
	p-Isopropyltoluene	5	ug/L
	Methylene chloride	5	ug/L
	Naphthalene	5	ug/L
	n-Propylbenzene	5	ug/L
Styrene	5	ug/L	
1,1,1,2-Tetrachloroethane	5	ug/L	
1,1,2,2-Tetrachloroethane	5	ug/L	
Tetrachloroethene	5	ug/L	
Toluene	5	ug/L	
1,2,3-Trichlorobenzene	5	ug/L	
1,2,4-Trichlorobenzene	5	ug/L	
1,1,1-Trichloroethane	5	ug/L	
1,1,2-Trichloroethane	5	ug/L	
Trichloroethene	5	ug/L	

Analyte	RL	Units
Trichlorofluoromethane	5	ug/L
1,2,3-Trichloropropane	5	ug/L
1,2,4-Trimethylbenzene	5	ug/L
1,3,5-Trimethylbenzene	5	ug/L
Vinyl chloride	5	ug/L
m-Xylene & p-Xylene	10	ug/L
o-Xylene	5	ug/L

---

**Surrogates**

4-Bromofluorobenzene  
1,2-Dichloroethane-d4  
Toluene-d8  
Dibromofluoromethane

**MS Volatiles – Method 8260B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
	Benzene	250	ug/kg
	Bromobenzene	250	ug/kg
	Bromochloromethane	250	ug/kg
	Bromodichloromethane	250	ug/kg
	Bromoform	250	ug/kg
	Bromomethane	250	ug/kg
	n-Butylbenzene	250	ug/kg
	sec-Butylbenzene	250	ug/kg
	tert-Butylbenzene	250	ug/kg
	Carbon tetrachloride	250	ug/kg
	Chlorobenzene	250	ug/kg
	Chlorodibromomethane	250	ug/kg
	Chloroethane	250	ug/kg
	Chloroform	250	ug/kg
	Chloromethane	250	ug/kg
	2-Chlorotoluene	250	ug/kg
	4-Chlorotoluene	250	ug/kg
	1,2-Dibromo-3-chloropropane	500	ug/kg
	1,2-Dibromoethane	250	ug/kg
	Dibromomethane	250	ug/kg
<b>Encore</b>	1,2-Dichlorobenzene	250	ug/kg
	1,3-Dichlorobenzene	250	ug/kg
	1,4-Dichlorobenzene	250	ug/kg
	Dichlorodifluoro-methane	250	ug/kg
	1,1-Dichloroethane	250	ug/kg
	1,2-Dichloroethane	250	ug/kg
	cis-1,2-Dichloroethene	250	ug/kg
	trans-1,2-Dichloroethene	250	ug/kg
	1,1-Dichloroethene	250	ug/kg
	1,2-Dichloropropane	250	ug/kg
	1,3-Dichloropropane	250	ug/kg
	2,2-Dichloropropane	250	ug/kg
	1,1-Dichloropropene	250	ug/kg
	Ethylbenzene	250	ug/kg
	Hexachlorobutadiene	250	ug/kg
	Isopropylbenzene	250	ug/kg
	p-Isopropyltoluene	250	ug/kg
	Methylene chloride	250	ug/kg
	Naphthalene	250	ug/kg
	n-Propylbenzene	250	ug/kg
	Styrene	250	ug/kg
	1,1,1,2-Tetrachloroethane	250	ug/kg
	1,1,2,2-Tetrachloroethane	250	ug/kg
	Tetrachloroethene	250	ug/kg
	Toluene	250	ug/kg
	1,2,3-Trichlorobenzene	250	ug/kg
	1,2,4-Trichlorobenzene	250	ug/kg
	1,1,1-Trichloroethane	250	ug/kg
	1,1,2-Trichloroethane	250	ug/kg

	Analyte	RL	Units
	Trichloroethene	250	ug/kg
	Trichlorofluoromethane	250	ug/kg
	1,2,3-Trichloropropane	250	ug/kg
	1,2,4-Trimethylbenzene	250	ug/kg
	1,3,5-Trimethylbenzene	250	ug/kg
	Vinyl chloride	250	ug/kg
	m-Xylene & p-Xylene	500	ug/kg
	o-Xylene	250	ug/kg

---

<b>Surrogates</b>	4-Bromofluorobenzene
	1,2-Dichloroethane-d4
	Toluene-d8
	Dibromofluoromethane

**MS Volatiles – Method 8260B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
<b>Low Level Encore</b>	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
	Styrene	5	ug/kg
	1,1,1,2-Tetrachloroethane	5	ug/kg
	1,1,2,2-Tetrachloroethane	5	ug/kg
	Tetrachloroethene	5	ug/kg
	Toluene	5	ug/kg
	1,2,3-Trichlorobenzene	5	ug/kg
	1,2,4-Trichlorobenzene	5	ug/kg
	1,1,1-Trichloroethane	5	ug/kg
	1,1,2-Trichloroethane	5	ug/kg
Trichloroethene	5	ug/kg	

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg

---

**Surrogates**

4-Bromofluorobenzene  
1,2-Dichloroethane-d4  
Toluene-d8  
Dibromofluoromethane

**MS Volatiles – Method 8260B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Frozen Encore</b>	Benzene	5	ug/kg
	Bromobenzene	5	ug/kg
	Bromochloromethane	5	ug/kg
	Bromodichloromethane	5	ug/kg
	Bromoform	5	ug/kg
	Bromomethane	5	ug/kg
	n-Butylbenzene	5	ug/kg
	sec-Butylbenzene	5	ug/kg
	tert-Butylbenzene	5	ug/kg
	Carbon tetrachloride	5	ug/kg
	Chlorobenzene	5	ug/kg
	Chlorodibromomethane	5	ug/kg
	Chloroethane	5	ug/kg
	Chloroform	5	ug/kg
	Chloromethane	5	ug/kg
	2-Chlorotoluene	5	ug/kg
	4-Chlorotoluene	5	ug/kg
	1,2-Dibromo-3-chloropropane	10	ug/kg
	1,2-Dibromoethane	5	ug/kg
	Dibromomethane	5	ug/kg
	1,2-Dichlorobenzene	5	ug/kg
	1,3-Dichlorobenzene	5	ug/kg
	1,4-Dichlorobenzene	5	ug/kg
	Dichlorodifluoro-methane	5	ug/kg
	1,1-Dichloroethane	5	ug/kg
	1,2-Dichloroethane	5	ug/kg
	cis-1,2-Dichloroethene	5	ug/kg
	trans-1,2-Dichloroethene	5	ug/kg
	1,1-Dichloroethene	5	ug/kg
	1,2-Dichloropropane	5	ug/kg
	1,3-Dichloropropane	5	ug/kg
	2,2-Dichloropropane	5	ug/kg
	1,1-Dichloropropene	5	ug/kg
	Ethylbenzene	5	ug/kg
	Hexachlorobutadiene	5	ug/kg
	Isopropylbenzene	5	ug/kg
	p-Isopropyltoluene	5	ug/kg
	Methylene chloride	5	ug/kg
	Naphthalene	5	ug/kg
	n-Propylbenzene	5	ug/kg
Styrene	5	ug/kg	
1,1,1,2-Tetrachloroethane	5	ug/kg	
1,1,2,2-Tetrachloroethane	5	ug/kg	
Tetrachloroethene	5	ug/kg	
Toluene	5	ug/kg	
1,2,3-Trichlorobenzene	5	ug/kg	
1,2,4-Trichlorobenzene	5	ug/kg	
1,1,1-Trichloroethane	5	ug/kg	
1,1,2-Trichloroethane	5	ug/kg	
Trichloroethene	5	ug/kg	

	Analyte	RL	Units
	Trichlorofluoromethane	5	ug/kg
	1,2,3-Trichloropropane	5	ug/kg
	1,2,4-Trimethylbenzene	5	ug/kg
	1,3,5-Trimethylbenzene	5	ug/kg
	Vinyl chloride	5	ug/kg
	m-Xylene & p-Xylene	10	ug/kg
	o-Xylene	5	ug/kg

---

<b>Surrogates</b>	4-Bromofluorobenzene		
	1,2-Dichloroethane-d4		
	Toluene-d8		
	Dibromofluoromethane		

**MS Volatiles – Method 624**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Benzene	5	ug/L
	Bromodichloro-methane	5	ug/L
	Bromoform	5	ug/L
	Bromomethane	55	ug/L
	Carbon tetrachloride	5	ug/L
	Chlorobenzene	5	ug/L
	Dibromochloro-methane	5	ug/L
	Chloroethane	5	ug/L
	2-Chloroethyl vinyl ether	--	ug/L
	Chloroform	5	ug/L
	Chloromethane	5	ug/L
	1,2-Dichlorobenzene	5	ug/L
	1,3-Dichlorobenzene	5	ug/L
	1,4-Dichlorobenzene	5	ug/L
	1,1-Dichloroethane	5	ug/L
	1,2-Dichloroethane	5	ug/L
	trans-1,2-Dichloroethene	5	ug/L
	1,1-Dichloroethene	5	ug/L
	1,2-Dichloropropane	5	ug/L
	cis-1,3-Dichloropropene	5	ug/L
	trans-1,3-Dichloropropene	5	ug/L
	Ethylbenzene	5	ug/L
	Methylene chloride	5	ug/L
	1,1,2,2-Tetrachloroethane	5	ug/L
	Tetrachloroethene	5	ug/L
	Toluene	5	ug/L
	1,1,1-Trichloroethane	5	ug/L
	1,1,2-Trichloroethane	5	ug/L
	Trichloroethene	5	ug/L
	Trichlorofluoro-methane	5	ug/L
Vinyl chloride	5	ug/L	
<b>Surrogates</b>	Bromofluorobenzene		
	1,2-Dichloro-ethane-d4		
	Toluene-d8		

**GC Semivolatiles – Method 8081A**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	alpha-Chlordane	0.05	ug/L
	gamma-Chlordane	0.05	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Endrin ketone	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	Methoxychlor	0.1	ug/L
Toxaphene	2	ug/L	
<b>Surrogates</b>	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

**GC Semivolatiles – 8081A**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Solid</b>	Aldrin	1.7	ug/kg
	alpha-BHC	1.7	ug/kg
	beta-BHC	1.7	ug/kg
	delta-BHC	1.7	ug/kg
	gamma-BHC (Lindane)	1.7	ug/kg
	alpha-Chlordane	1.7	ug/kg
	gamma-Chlordane	1.7	ug/kg
	4,4'-DDD	1.7	ug/kg
	4,4'-DDE	1.7	ug/kg
	4,4'-DDT	1.7	ug/kg
	Dieldrin	1.7	ug/kg
	Endosulfan I	1.7	ug/kg
	Endosulfan II	1.7	ug/kg
	Endosulfan sulfate	1.7	ug/kg
	Endrin	1.7	ug/kg
	Endrin aldehyde	1.7	ug/kg
	Endrin ketone	1.7	ug/kg
	Heptachlor	1.7	ug/kg
	Heptachlor epoxide	1.7	ug/kg
	Methoxychlor	3.3	ug/kg
Toxaphene	67	ug/kg	
<b>Surrogates</b>	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

**GC Semivolatiles – Method 8082**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Aroclor 1016	1	ug/L
	Aroclor 1221	1	ug/L
	Aroclor 1232	1	ug/L
	Aroclor 1242	1	ug/L
	Aroclor 1248	1	ug/L
	Aroclor 1254	1	ug/L
	Aroclor 1260	1	ug/L
	Aroclor 1262	1	ug/L
	Aroclor 1268	1	ug/L
<b>Surrogates</b>	Decachlorobi-phenyl Tetrachloro-m-xylene		
<b>Solid</b>	Aroclor 1016	33	ug/kg
	Aroclor 1221	33	ug/kg
	Aroclor 1232	33	ug/kg
	Aroclor 1242	33	ug/kg
	Aroclor 1248	33	ug/kg
	Aroclor 1254	33	ug/kg
	Aroclor 1260	33	ug/kg
	Aroclor 1262	33	ug/kg
	Aroclor 1268	33	ug/kg
<b>Surrogates</b>	Decachloro-biphenyl Tetrachloro-m-xylene		

**GC Semivolatiles – Method 608**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Aldrin	0.05	ug/L
	alpha-BHC	0.05	ug/L
	beta-BHC	0.05	ug/L
	delta-BHC	0.05	ug/L
	gamma-BHC (Lindane)	0.05	ug/L
	Chlordane (technical)	0.5	ug/L
	4,4'-DDD	0.05	ug/L
	4,4'-DDE	0.05	ug/L
	4,4'-DDT	0.05	ug/L
	Dieldrin	0.05	ug/L
	Endosulfan I	0.05	ug/L
	Endosulfan II	0.05	ug/L
	Endosulfan sulfate	0.05	ug/L
	Endrin	0.05	ug/L
	Endrin aldehyde	0.05	ug/L
	Heptachlor	0.05	ug/L
	Heptachlor epoxide	0.05	ug/L
	PCB-1016	1	ug/L
	PCB-1221	1	ug/L
	PCB-1232	1	ug/L
PCB-1242	1	ug/L	
PCB-1248	1	ug/L	
PCB-1254	1	ug/L	
PCB-1260	1	ug/L	
Toxaphene	2	ug/L	
<b>Surrogates</b>	Decachloro-biphenyl		
	Tetrachloro-m-xylene		

**GC Semivolatiles – Method 8151A**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	2,4-D	4	ug/L
	Dalapon	2	ug/L
	2,4-DB	4	ug/L
	Dicamba	2	ug/L
	Dichlorprop	4	ug/L
	Dinoseb	0.6	ug/L
	MCPA	400	ug/L
	MCPP	400	ug/L
	2,4,5-TP (Silvex)	1	ug/L
	2,4,5-T	1	ug/L
<b>Surrogate</b>	2,4-Dichlorophenylacetic acid		
<b>Solid</b>	2,4-D	80	ug/kg
	Dalapon	40	ug/kg
	2,4-DB	80	ug/kg
	Dicamba	40	ug/kg
	Dichlorprop	80	ug/kg
	Dinoseb	12	ug/kg
	MCPA	8000	ug/kg
	MCPP	8000	ug/kg
	2,4,5-TP (Silvex)	20	ug/kg
	2,4,5-T	20	ug/kg
<b>Surrogate</b>	2,4-Dichlorophenylacetic acid		

**GC Volatiles - Method 8021B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b> <b>5 mL Purge</b>	Benzene	1	ug/L
	Bromobenzene	1	ug/L
	Bromochloromethane	1	ug/L
	Bromodichloromethane	1	ug/L
	Bromoform	1	ug/L
	Bromomethane	1	ug/L
	n-Butylbenzene	1	ug/L
	sec-Butylbenzene	1	ug/L
	tert-Butylbenzene	1	ug/L
	Carbon tetrachloride	1	ug/L
	Chlorobenzene	1	ug/L
	Chlorodibromomethane	1	ug/L
	Chloroethane	1	ug/L
	Chloroform	1	ug/L
	Chloromethane	1	ug/L
	2-Chlorotoluene	1	ug/L
	4-Chlorotoluene	1	ug/L
	1,2-Dibromo-3-chloropropane (DBCP)	1	ug/L
	1,2-Dibromoethane (EDB)	1	ug/L
	Dibromomethane	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Dichlorodifluoromethane	1	ug/L
	1,1-Dichloroethane	1	ug/L
	1,2-Dichloroethane	1	ug/L
	cis-1,2-Dichloroethene	1	ug/L
	trans-1,2-Dichloroethene	1	ug/L
	1,1-Dichloroethene	1	ug/L
	1,2-Dichloropropane	1	ug/L
	1,3-Dichloropropane	1	ug/L
	2,2-Dichloropropane	1	ug/L
	cis-1,3-Dichloropropene	1	ug/L
	trans-1,3-Dichloropropene	1	ug/L
	1,1-Dichloropropene	1	ug/L
	Ethylbenzene	1	ug/
	Hexachlorobutadiene	1	ug/L
	Isopropylbenzene	1	ug/L
	p-Isopropyltoluene	1	ug/L
	Methylene chloride	5	ug/L
Naphthalene	1	ug/L	
n-Propylbenzene	1	ug/L	
Styrene	1	ug/L	
1,1,1,2-Tetrachloroethane	1	ug/L	
1,1,2,2-Tetrachloroethane	1	ug/L	
Tetrachloroethene	1	ug/L	
Toluene	1	ug/L	
1,2,3-Trichlorobenzene	1	ug/L	
1,2,4-Trichlorobenzene	1	ug/L	

**GC Volatiles - Method 8021B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

Analyte	RL	Units
1,1,1-Trichloroethane	1	ug/L
1,1,2-Trichloroethane	1	ug/L
Trichloroethene	1	ug/L
Trichlorofluoromethane	1	ug/L
1,2,3-Trichloropropane	1	ug/L
1,2,4-Trimethylbenzene	1	ug/L
1,3,5-Trimethylbenzene	1	ug/L
Vinyl chloride	1	ug/L
m-Xylene & p-Xylene	2	ug/L
o-Xylene	1	ug/L
Xylenes (total)	3	ug/L

---

<b>Surrogates</b>	1,4-Dichlorobutane
	Trifluorotoluene

**GC Volatiles - Method 8021B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Solid 5 g</b>	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg
	<b>Surrogates</b>	Trifluorotoluene	

**GC Volatiles – Method 8021B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Solid Encore, Methanol Prep</b>	Benzene	50	ug/kg
	Ethylbenzene	50	ug/kg
	Methyl tert-butyl ether (MTBE)	50	ug/kg
	Naphthalene	250	ug/kg
	Toluene	50	ug/kg
	1,2,4-Trimethylbenzene	50	ug/kg
	1,3,5-Trimethylbenzene	50	ug/kg
	m-Xylene & p-Xylene	100	ug/kg
	o-Xylene	50	ug/kg
	Xylenes (total)	150	ug/kg
<b>Surrogates</b>	Trifluorotoluene		

**GC Volatiles – Method 8021B**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Frozen Encore</b>	Benzene	1	ug/kg
	Ethylbenzene	1	ug/kg
	Methyl tert-butyl ether (MTBE)	1	ug/kg
	Methyl tert-butyl ether	1	ug/kg
	Naphthalene	1	ug/kg
	Toluene	1	ug/kg
	1,2,4-Trimethylbenzene	1	ug/kg
	1,3,5-Trimethylbenzene	1	ug/kg
	m-Xylene & p-Xylene	2	ug/kg
	o-Xylene	1	ug/kg
	Xylenes (total)	3	ug/kg

---

**Surrogates**                      Trifluorotoluene

**GC Volatiles – Method 601**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units	
<b>Water</b>	Bromodichloromethane	1	ug/L	
	Bromoform	1	ug/L	
	Bromomethane	1	ug/L	
	Carbon tetrachloride	1	ug/L	
	Chlorobenzene	1	ug/L	
	Dibromochloromethane	1	ug/L	
	Chloroethane	1	ug/L	
	2-Chloroethyl vinyl ether	5	ug/L	
	Chloroform	1	ug/L	
	Chloromethane	1	ug/L	
	1,2-Dichlorobenzene	1	ug/L	
	1,3-Dichlorobenzene	1	ug/L	
	1,4-Dichlorobenzene	1	ug/L	
	Dichlorodifluoromethane	1	ug/L	
	1,1-Dichloroethane	1	ug/L	
	1,2-Dichloroethane	1	ug/L	
	trans-1,2-Dichloroethene	1	ug/L	
	1,1-Dichloroethene	1	ug/L	
	1,2-Dichloropropane	1	ug/L	
	cis-1,3-Dichloropropene	1	ug/L	
	trans-1,3-Dichloropropene	1	ug/L	
	Methylene chloride	5	ug/L	
	1,1,2,2-Tetrachloroethane	1	ug/L	
	Tetrachloroethene	1	ug/L	
	1,1,1-Trichloroethane	1	ug/L	
	1,1,2-Trichloroethane	1	ug/L	
	Trichloroethene	1	ug/L	
	Trichlorofluoromethane	1	ug/L	
	Vinyl chloride	1	ug/L	
	<b>Surrogates</b>	1,4-Dichlorobutane		

**GC Volatiles – Method 602**  
**Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Water</b>	Benzene	1	ug/L
	Chlorobenzene	1	ug/L
	1,2-Dichlorobenzene	1	ug/L
	1,3-Dichlorobenzene	1	ug/L
	1,4-Dichlorobenzene	1	ug/L
	Ethylbenzene	1	ug/L
	Toluene	1	ug/L
<b>Surrogates</b>	Trifluorotoluene		

**Total Petroleum Hydrocarbons, Diesel & Gasoline Range Organics  
 Reporting Limits (RL), Method Reference <sup>1,2</sup>**

	Analyte	RL	Units
<b>Diesel Range Organics - Water</b>	TPH (as Diesel)	100	ug/L
<b>Surrogate</b>	C9 (nonane)		
<b>Diesel Range Organics - Solid</b>	TPH (as Diesel)	10	mg/kg
<b>Surrogate</b>	C9 (nonane)		
<b>Gasoline Range Organics - Water</b>	Gasoline Range Organics	100	ug/L
	TPH (as Gasoline)	100	ug/L
	TPH (Purgeables)	100	ug/L
<b>Gasoline Range Organics - Solid</b>	Gasoline Range Organics	100	ug/kg
	TPH (as Gasoline)	100	ug/kg
	TPH (Purgeables)	100	ug/kg
<b>Surrogate</b>	TFT		
<b>Gasoline Range Organics – Medium Level</b>	TPH (as Gasoline) Solid	1000	ug/kg
	TPH (as Gasoline)	1000	ug/kg
	TPH (Purgeables)	1000	ug/kg
<b>Surrogate</b>	TFT		

Footnote:

<sup>1</sup> The latest MDLs, RLs, and Control Limits will be utilized at the time of sample analysis, and are available upon request.

<sup>2</sup> The compounds listed represent TAL/TCL compounds. Project specific or other regulatory lists may be available upon request.

## **Appendix 6. 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)

**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 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 quantitative and qualitative specifications of some operational function or activity. (EPA-QAD)

**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)

**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 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:**

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 is 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 Insecticide, Fungicide and Rodenticide Act (FIFRA):**

The enabling legislation under 7 U.S.C. 135 et seq., as amended, that empowers the EPA to register insecticides, fungicides, and rodenticides. (NELAC)

**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 filling 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)

**Finding:**

An assessment conclusion that identifies a condition having a significant effect on an item or activity. An assessment finding is normally a deficiency and is normally accompanied by specific examples of the observed condition. (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)

**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)

**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)

**Instrument Response:**

Instrument response is normally expressed as either peak area or peak height however it may also reflect a numerical representation of some type of count on a detector (e.g. Photomultiplier tube, or Diode array detector) and is used in this document to represent all types.

**Laboratory:**

A defined facility performing environmental analyses in a controlled and scientific manner. (NELAC)

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.

Manager (however named):

The individual designed as being responsible 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 supervisor and the manager may be the same individual. (NELAC)

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 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)

**National Environmental Laboratory Accreditation Conference (NELAC):**

A voluntary organization of State and Federal environmental officials and interest groups purposed primarily to establish mutually acceptable standards for accrediting environmental laboratories. A subset of NELAP. (NELAC)

**National Environmental Laboratory Accreditation Program (NELAP):**

The overall National Environmental Laboratory Accreditation Program of which NELAC is a part. (NELAC)

**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)

**NELAC Standards:**

The plan of procedures for consistently evaluating and documenting the ability of laboratories performing environmental measurements to meet nationally defined standards established by the National Environmental Laboratory Accreditation Conference. (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 Method:**

A method of known and documented accuracy and precision issued by an organization recognized as competent to do so. (NELAC)

**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)

**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)

**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<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)

**Supervisor (however named):**

The individual(s) designated as being responsible for a particular area or category of scientific analysis. This responsibility includes direct day-to-day supervision of technical employees, supply and instrument adequacy and upkeep, quality assurance/quality control duties, and ascertaining that technical employees have the required balance of education, training and experience to perform the required analyses. (NELAC)

**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)

**Technical Director:**

Individuals(s) who has overall responsibility for the technical operation of the environmental testing laboratory. (NELAC)

**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 a specific measurement problem, as documented in a laboratory SOP. (NELAC)

**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.

United States Environmental Protection Agency (EPA):

The Federal governmental agency with responsibility for protecting public health and safeguarding and improving the natural environment (i.e., the air, water, and land) upon which human life depends. (US-EPA)

Validation:

The process of substantiating specified performance criteria. (EPA-QAD)

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 adjustment, to repair, 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.

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)

## **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 7.

### Laboratory Certifications, Accreditations, Validations

TestAmerica North Canton 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 Number	Organization	Certificate Number
California	01144CA	New Jersey	OH001
Connecticut	PH-0590	New York	10975
Florida	E87225	Pennsylvania	68-00340
Georgia	--	OVAP	CL0024
Illinois	200004	US ACE (Army)	--
Kansas	E-10336	USDA (Dept.of Agriculture)	S-62345
Kentucky Underground Storage Tank Program	0058	West Virginia	210
Minnesota	039-999-348	Wisconsin	999518190

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.

### Claims of Accreditation Status

TestAmerica North Canton has agreed to make only valid claims as to its accreditation/certification status by any authority by ensuring that the expiration dates are not exceeded and the method-specific scope or parameter lists are supportable, as required by each. Any false claims would be reported to that authority. The agreement covers the use of the authority's name, such as "Authority-Accredited," logo, or certificate number. The only valid proof of accreditation/certification is the current certificate and scope of the authority. It is the responsibility of the laboratory to make these documents available to all staff, and it is the staff's duty to reference only the current documents.

A report with scope and non-scope analytes may only be presented on the same report if the non-accredited results are clearly and unambiguously identified. No report with non-scope analytes may be associated with the logo, "Authority accredited" phrase, or the certificate number. Only the analytes specified by a unique method are valid within the scope. There shall be no intentional misleading of the users of the laboratory's services in this regard.

No opinions and/or interpretations based on results outside the laboratory's scope may be presented on a document referenced by "Authority-accredited, the logo, or the certificate number. If these are made, they must be written in a separate letter which is not endorsed by the authority.

The "Authority-accredited" logo may only be affixed to equipment calibrated by a laboratory that is accredited by the authority. If calibration labels contain the logo, they must also show the calibration laboratory's name or its certificate number, the instrument's unique identification, the date of the last calibration, and a cross-reference to the last calibration certificate.

Should the company decide to use the "Authority-accredited" logo in marketing activities, no misrepresentation may occur. Only reference to the accredited scope at a specific laboratory site is allowed. If any "Authority-accredited" language is used in proposals or quotations, any non-scope analytes must be clearly denoted as not accredited by that authority. The same is true for any use of laboratory letterhead with the "Authority-accredited" wording or logo. The logo may not be affixed to any material, item, product, part, or packaging, thereby implying accreditation status to that piece. In literature, any use of the logo must be positioned adjacent to the accredited laboratory's name and clearly state that the presence of the logo does not imply certification/approval of the products tested. At no time may the logo appear to suggest that a person is accredited. Misrepresentation of accreditation status is never allowed and must be reported if it occurs. If in doubt, the idea of the logo's use may be presented to the authority for approval.

If accreditation is terminated or suspended, the laboratory will immediately cease to use the "Authority-accredited" wording, the logo, or the certificate number reference in any way and inform clients impacted by the change.

**Appendix 8.**

**Data Qualifiers**

<b>Qualifier Organic</b>	<b>Qualifier Inorganic</b>	<b>Footnote</b>
U	U	Analyte analyzed for but was not detected.
G	G	Elevated reporting limit. The reporting limit is elevated due to matrix interference.
J	B	Estimated result. Result is less than RL.
E	I	Estimated result. Result concentration exceeds the calibration range.
M	M	Matrix spike recovery outside of limits.
B	J	Method blank contamination. The associated method blank contains the target analyte at a reportable level.
P	*	Relative percent difference (RPD) is outside stated control limits.
a	N	Spiked analyte recovery is outside stated control limits.
*		Surrogate recovery is outside stated control limits.
PG		The percent difference between the original and confirmation analyses is greater than 40%.

This is not an exhaustive list of qualifiers. All qualifiers are defined on each data sheet. Client specific qualifiers may also be used, and would also be defined on the data sheet.

## Title: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8260B AND 8260A

[ Method: EPA Methods 8260B AND 8260A ]

### Approvals (Signature/Date):

<u>Thomas E. Stiller</u>	<u>01/02/09</u>	<u>[Signature]</u>	<u>1/02/09</u>
Technology Specialist	Date	Health & Safety Coordinator	Date

<u>Dorothy J. Kuson</u>	<u>1/2/09</u>	<u>Mark L. Bane</u>	<u>1/2/09</u>
Quality Assurance Manager	Date	Technical Director	Date

<u>[Signature]</u>	<u>1/5/09</u>
Laboratory Director	Date

This SOP was previously identified as SOP No. NC-MS-019, Rev 0, dated 06/30/08

### Copyright Information:

This documentation has been prepared by TestAmerica Analytical Testing Corp. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY: ©COPYRIGHT 2008 TESTAMERICA ANALYTICAL TESTING CORP. ALL RIGHTS RESERVED.

Facility Distribution No. \_\_\_\_\_

Distributed To: \_\_\_\_\_

**TABLE OF CONTENTS**

**1. SCOPE AND APPLICATION ..... 4**

**2. SUMMARY OF METHOD ..... 4**

**3. DEFINITIONS..... 5**

**4. INTERFERENCES ..... 6**

**5. SAFETY ..... 7**

**6. EQUIPMENT AND SUPPLIES ..... 9**

**7. REAGENTS AND STANDARDS..... 11**

**8. SAMPLE COLLECTION, PRESERVATION AND STORAGE ..... 12**

**9. QUALITY CONTROL ..... 19**

**10. CALIBRATION AND STANDARDIZATION ..... 22**

**11. PROCEDURE..... 26**

**12. DATA ANALYSIS AND CALCULATIONS..... 31**

**13. METHOD PERFORMANCE..... 37**

**14. POLLUTION PREVENTION ..... 38**

**15. WASTE MANAGEMENT..... 38**

**16. REFERENCES ..... 38**

**17. MISCELLANEOUS..... 40**

## **LIST OF TABLES**

<b>Table 1</b>	<b>TestAmerica Primary Standard and Reporting Limits</b>
<b>Table 2</b>	<b>TestAmerica Primary Standard Calibration Levels</b>
<b>Table 2A</b>	<b>TestAmerica Primary Standard Calibration Levels- Low Level</b>
<b>Table 3</b>	<b>TestAmerica Appendix IX Standard and Reporting Limits</b>
<b>Table 4</b>	<b>TestAmerica Appendix IX Standard Calibration Levels</b>
<b>Table 5</b>	<b>Reportable Analytes for TestAmerica Standard Tests, Primary Standard</b>
<b>Table 6</b>	<b>Reportable Analytes for TestAmerica Standard Tests, Appendix IX Standard</b>
<b>Table 7</b>	<b>Internal Standards</b>
<b>Table 8</b>	<b>Surrogate Standards</b>
<b>Table 9</b>	<b>Matrix Spike / LCS Control Standards</b>
<b>Table 10</b>	<b>BFB Tune Criteria</b>
<b>Table 11</b>	<b>SPCC Compounds and Minimum Response Factors</b>
<b>Table 12</b>	<b>CCC Compounds</b>
<b>Table 13</b>	<b>Characteristic Ions</b>

## 1.0 SCOPE AND APPLICATION

- 1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewater, soils, sludges, and other solid matrices. Standard analytes are listed in Tables 5 and 6.
- 1.2.1. This SOP is applicable to Method 8260B. It may also be used for analysis following Method 8260A. The associated LIMS method codes are QK (8260B) and MZ (8260A). Ohio VAP projects are distinguished by Program Code 2J. The following Prep Codes are used: 15 (5 mL purge), 25 (low-level 5mL purge), 4B (5035, Methanol Preservation, EnCore™), 4D (5035, Sodium Bisulfate Preservation, EnCore™), 4P (Frozen, EnCore™), M8 (5035A, Frozen Encore™), and 73 (5030A Methanol Prep).
- 1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water-soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.
- 1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 40 µg/L for low-level waters, 5 to 200 µg/kg for low-level soils, and 250 to 10,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1 and 3.
- 1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

## 2.0 SUMMARY OF METHOD

- 2.1. Volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer, which is used to provide both qualitative and quantitative information.
- 2.2. Aqueous samples are purged directly. Soils are preserved by extracting the volatile analytes into methanol. Soil samples may be preserved with sodium bisulfate or by freezing and purging directly.
- 2.3. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature or at 40°C (40°C required for low level soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components, which are detected with a mass spectrometer.

- 2.4. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

### **3.0 DEFINITIONS**

#### **3.1. Batch**

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents 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.

- 3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. Refer to the TestAmerica QC program document (QA-003) for further details of the batch definition.

#### **3.2. Method Blank**

- 3.2.1. A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

#### **3.3. Laboratory Control Sample (LCS)**

- 3.3.1. Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

#### **3.4. Surrogates**

- 3.4.1. Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within

the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

3.5.1. A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample, which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

3.6.1. CCCs are a representative group of compounds, which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

3.7.1. SPCCs are compounds, which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

#### 4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. All glassware is cleaned per SOP NC-QA-014. The use of ultra high purity gases, prepurged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases, the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination. Refer to SOP NC-QA-020 for additional information on holding blanks.

4.3. Matrix interferences may be caused by non-target contaminants that are co-extracted from

the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

- 4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it must be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.
- 4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered, the sample must be diluted.

## 5. SAFETY

- 5.1 Employees must abide by the policies and procedures in the Corporate Environmental Health and Safety Manual and this document.
- 5.2 Eye protection that protects against splash, laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated must be removed and discarded; other gloves must be cleaned immediately. Cut-resistant gloves **MUST** be worn when opening VOA vials and when doing any other task that presents a strong possibility of getting cut.
- 5.3 Primary Materials Used
  - 5.3.1 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 Standards 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
--------------	---------	--------------------	--------------------------------

Sodium bisulfate	Irritant	None	Causes mild to severe irritation to the eyes. Prolonged exposure may cause burn if not flushed with water. May cause mild irritation to skin. Prolonged exposure may cause burn if not flushed with water.
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.
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 – Always add acid to water to prevent violent reactions.			
2 – Exposure limit refers to the OSHA regulatory exposure limit.			

- 5.4. It is recommended that analysts break up work tasks to avoid repetitive motion tasks, such as opening a large number of vials or containers in one time period.
- 5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**. All samples with a sticker that reads "Caution/Use Hood!" **must** be opened in the hood. Contact the EH&S Coordinator if this is not possible. Solvent and waste containers must be kept closed unless transfers are being made. MS VOA samples may be prepared outside of the hood, unless it is known that concentrations are high.
- 5.6. The preparation of standards and reagents must be conducted in a fume hood with the sash closed as far as the operations will permit. MS VOA standards may be prepared outside of the hood due to low concentrations of analytes.
- 5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a TestAmerica associate. The situation must be reported **immediately** to the EH&S Coordinator and the laboratory Group Leader.
- 5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the TestAmerica Corporate Environmental Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed

by an annual refresher training.

#### 5.9. Specific Safety Concerns or Requirements

5.9.1. 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.

5.9.2. The mass spectrometer is under deep vacuum. The mass spectrometer must be brought to atmospheric pressure prior to working on the source.

5.9.3. 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.

5.9.4. Sodium bisulfate creates Sulfuric Acid when mixed with water.

## 6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10  $\mu$ L and larger

6.2. Syringe: 5, 25, or 50 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001g, and a top-loading balance capable of weighing 0.1g

6.4. Glassware:

6.4.1. Vials: 20 and 40 mL with screw caps and Teflon® liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipettes: Pasteur, 5  $\frac{3}{4}$  in.

6.7. pH paper: Wide range, pH 0-14.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Nitrogen: Ultra high purity from cylinders or gas generators may be used as an alternative to helium for purge gas.

- 6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, trap, and desorber.
- 6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated. Low-level soils are purged directly from a VOA vial.
- 6.9.2. Trap: A variety of traps may be used, depending on the target analytes required. One of the traps used is the Vocarb 3000 trap. Other traps such as the OI 10 may be used if the Quality Control criteria are met. Refer also to instrument operating manuals located within the laboratory.
- 6.9.3. Desorber: The desorber must be capable of rapidly heating the trap to at least 180°C. Many such devices are commercially available.
- 6.9.4. Sample Heater: A heater capable of maintaining the purge device at 40°C is necessary for low-level soil analysis.
- 6.10. Gas Chromatograph/Mass Spectrometer System:
- 6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming.
- 6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:
- 6.10.2.1. Column 1: 20m x 0.18 ID DB-624 with 1 µm film thickness.
- 6.10.2.2. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.
- 6.10.3. GC/MS Interface: In general direct introduction to the mass spectrometer is used but any interface that achieves all acceptance criteria may be used.
- 6.10.4. Data System: A computer system that allows the continuous acquisition and storage on machine-readable media of all mass spectra obtained throughout the

duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The NIST/EPA mass spectral library must be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

## 7. REAGENTS AND STANDARDS

### 7.1. Reagents

7.1.1. Methanol: Purge and Trap grade, high purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed (see Section 9.3). Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.1.3. Hydrochloric Acid – (1:1 v/v): Reagent grade or equivalent

7.1.4. Sodium bisulfate: Reagent grade or equivalent

### 7.2. Standards

#### 7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon®-sealed screw-cap bottles with minimal headspace at -10° to -20°C.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored in the freezer or as recommended by the manufacturer. Working standards are monitored by comparison to the initial calibration curve. If any of the calibration check compounds drift in response from the initial calibration by more than 20% then corrective action is necessary. This may include steps such as instrument maintenance, preparing a new calibration verification standard or tuning the instrument. If the corrective actions do not correct the problem then a new initial calibration must be performed.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules, they may be used up to the manufacturer's expiration date.

7.2.1.5. Additional information can be found in SOP NC-QA-017.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 50 ng/ $\mu$ L of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

7.2.7. All standard preparation information is detailed in the Standard Logbook.

## **8. SAMPLE COLLECTION, PRESERVATION, AND STORAGE**

8.1. Holding times for all volatile analysis are 14 days from sample collection to analysis.

8.2. For DoD samples, water samples are normally preserved at  $\text{pH} \leq 2$  with 1:1 hydrochloric acid. Unpreserved water samples must be analyzed within seven days of sampling.

8.3. Solid samples are field preserved with sodium bisulfate solution or by freezing upon receipt at the laboratory for low-level analysis, or with methanol for medium-level analysis. Soil samples can also be taken using the EnCore™ sampler and preserved in the lab within 48 hours of sampling. Analysis must be completed 14 days from sampling. At specific client request, unpreserved soil samples may be accepted.

8.4. There are several methods of sampling soil. The recommended method, which provides the minimum of field difficulties, is to take an EnCore™ sample. (The 5g or 25g sampler can be used, depending on client preference). Following shipment back to the lab, the soil is preserved in methanol. This is the medium level procedure. If very low detection limits are needed ( $< 50 \mu\text{g}/\text{kg}$  for most analytes), then it will be necessary to use two additional 5g

EnCore™ samplers or to use field preservation.

- 8.5. Sample collection for medium level analysis using EnCore™ samplers.
  - 8.5.1. Ship one 5g (or 25g) EnCore™ sampler per field sample position.
  - 8.5.2. An additional 2 oz plastic bottle must be shipped for percent moisture determination.
  - 8.5.3. When the samples are returned to the lab, extrude the (nominal) 5g (or 25g) sample into a tared VOA vial containing 5 mL methanol (25 mL methanol for the 25g sampler). Obtain the weight of the soil added to the vial and note on the label.
  - 8.5.4. Add the correct amount of surrogate spiking mixture. (Add 25 µL of 2500 µg/mL solution for a nominal 25g sample, 5 µL for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.
  - 8.5.5. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500 µL of 50 µg/mL solution for a nominal 25 g sample, 100 µL for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.
  - 8.5.6. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500 µL of spike to 25 mL methanol or 100 µL spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.
  - 8.5.7. Shake the samples for two minutes to distribute the methanol throughout the soil.
- 8.6. Sample collection for medium-level analysis using field methanol preservation
  - 8.6.1. Prepare a 2-oz sample container by adding 25 mL purge and trap grade methanol. (If a 5g sample is to be used, add 5 mL methanol to a 2 oz container or VOA vial).
  - 8.6.2. Seal the bottle and attach a label.
  - 8.6.3. Weigh the bottle to the nearest 0.01g, and note the weight on the label.
  - 8.6.4. Ship with appropriate sampling instructions.
  - 8.6.5. Each sample will require an additional 2 oz plastic bottle with no preservative

for percent moisture determination.

- 8.6.6. At client request, the methanol addition and weighing may also be performed in the field.
  - 8.6.7. When the samples are returned to the lab, obtain the weight of the soil added to the vial and note on the label.
  - 8.6.8. Add the correct amount of surrogate spiking mixture. (Add 25  $\mu\text{L}$  of 2500  $\mu\text{g}/\text{mL}$  solution for a nominal 25g sample, 5  $\mu\text{L}$  for a nominal 5g sample.) Refer to Section 17.2 for Michigan project criteria.
  - 8.6.9. Add the correct amount of matrix spiking solution to the matrix spike and matrix spike duplicate samples. (Add 500  $\mu\text{L}$  of 50  $\mu\text{g}/\text{mL}$  solution for a nominal 25g sample, 100  $\mu\text{L}$  for a nominal 5g sample.) Reduce the volume of methanol added to ensure the final volume is 25 mL for nominal 25g sample or 5 mL methanol for a nominal 5g sample. Refer to Section 17.2 for Michigan project criteria.
  - 8.6.10. Prepare an LCS for each batch by adding the correct amount of matrix spiking solution to clean methanol. (500  $\mu\text{L}$  of spike to 25 mL methanol or 100  $\mu\text{L}$  spike to 5 mL methanol). Refer to Section 17.2 for Michigan project criteria.
  - 8.6.11. Shake the samples for two minutes to distribute the methanol throughout the soil.
- 8.7. Low-level procedure
- 8.7.1. If low detection limits are required (typically  $< 50 \mu\text{g}/\text{kg}$ ), low-level soil preservation must be used. However, it is also necessary to take a sample for the medium-level (field methanol preserved or using the EnCore™ sampler) procedure in case the concentration of analytes in the soil is above the calibration range of the low-level procedure.
  - 8.7.2. A purge and trap autosampler capable of sampling from a sealed vial is required for analysis of samples collected using this method. (Varian Archon or O.I. 4552).
  - 8.7.3. The soil sample is taken using a 5g EnCore™ sampling device and returned to the lab. It is recommended that two EnCore™ samplers be used for each field sample position to allow for any reruns than may be necessary. A separate sample for % moisture determination is also necessary.
  - 8.7.4. Prepare VOA vials for sodium bisulfate preservation by adding a magnetic stir bar, approximately 1g of sodium bisulfate, and 5 mL of reagent water. Prepare vials for preservation by freezing by adding a stir bar and 5 mL reagent water.

- 8.7.5. Seal and label the vial. It is strongly recommended that the vial is labeled with an indelible marker rather than a paper label, since paper labels may cause the autosampler to bind and malfunction. The label absolutely must not cover the neck of the vial or the autosampler will malfunction.
- 8.7.6. Weigh the vial to the nearest 0.1g, and note the weight on the label.
- 8.7.7. Extrude the soil sample from the EnCore™ sampler into the prepared VOA vial. Reweigh the vial to obtain the weight of soil, and note on the label.

**Note:** Soils containing carbonates may effervesce when added to the sodium bisulfate solution. If this is the case at a specific site, add 5 mL of water instead, and freeze at <-10°C within 48 hours. The sample must be analyzed within 14 days after sampling and stored at a 45-degree angle in the freezer.

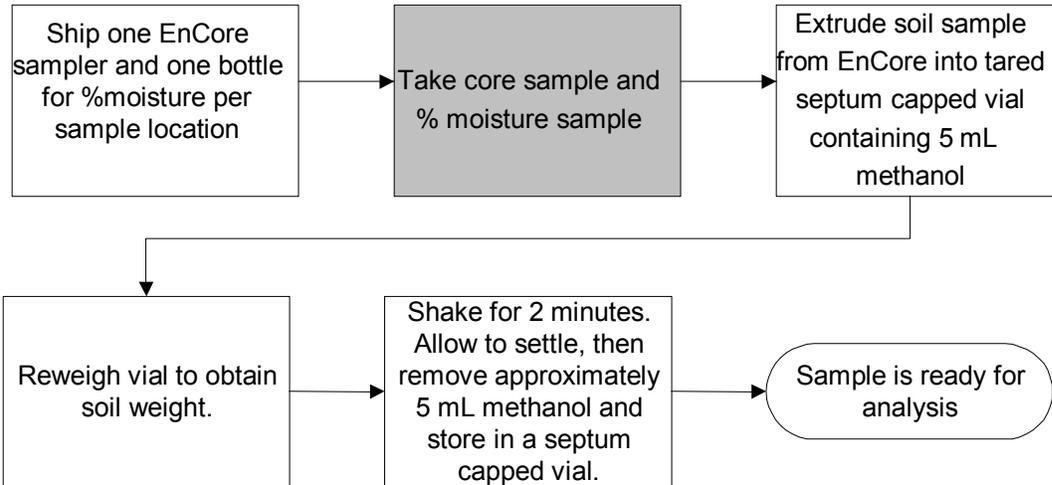
- 8.7.8. Alternatively the sodium bisulfate preservation may be performed in the field. This is not recommended because of the many problems that can occur in the field setting. Ship at least two vials per sample. The field samplers must determine the weight of soil sampled. Each sample will require an additional 2 oz plastic bottle with no preservative for percent moisture determination, and an additional VOA vial preserved with methanol for the medium level procedure. Depending on the type of soil, it may also be necessary to ship vials with no or extra preservative.

#### 8.8. *Unpreserved soils*

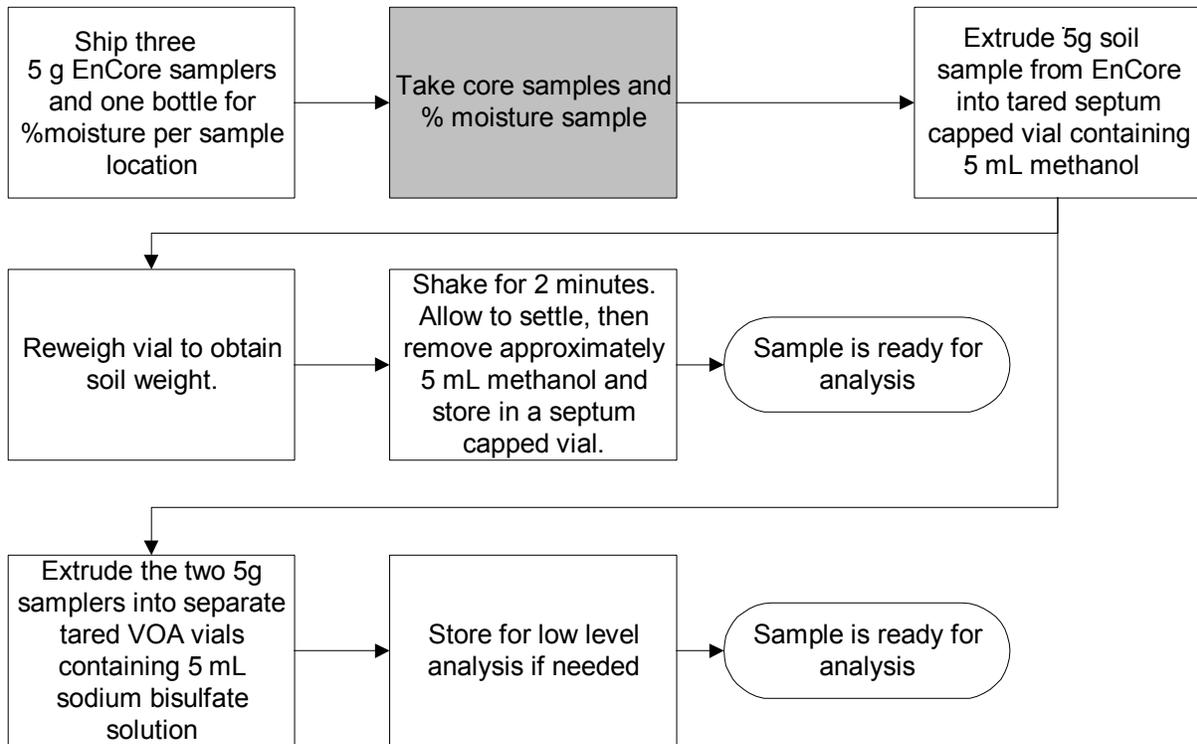
8.8.1. *At specific client request, unpreserved soils packed into glass jars or brass tubes may be accepted and sub-sampled in the lab. This is the old procedure based on Method 5030A and Method 8260A. It is no longer included in SW846 and is likely to generate results that are biased low, possibly by more than an order of magnitude.*

- 8.9. Aqueous samples are stored in glass containers with Teflon®-lined septa at 4°C +/- 2°C with minimum headspace.
- 8.10. The maximum holding time is 14 days from sampling until the sample is analyzed. (Samples that are found to be unpreserved still have a 14-day holding time. However, they should be analyzed as soon as possible. The lack of preservation must be addressed in the case narrative). Maximum holding time for the EnCore™ sampler (before the sample is added to methanol or sodium bisulfate) is 48 hours.
- 8.11. A holding blank is stored with the samples. This is analyzed weekly. It is replaced every seven days.

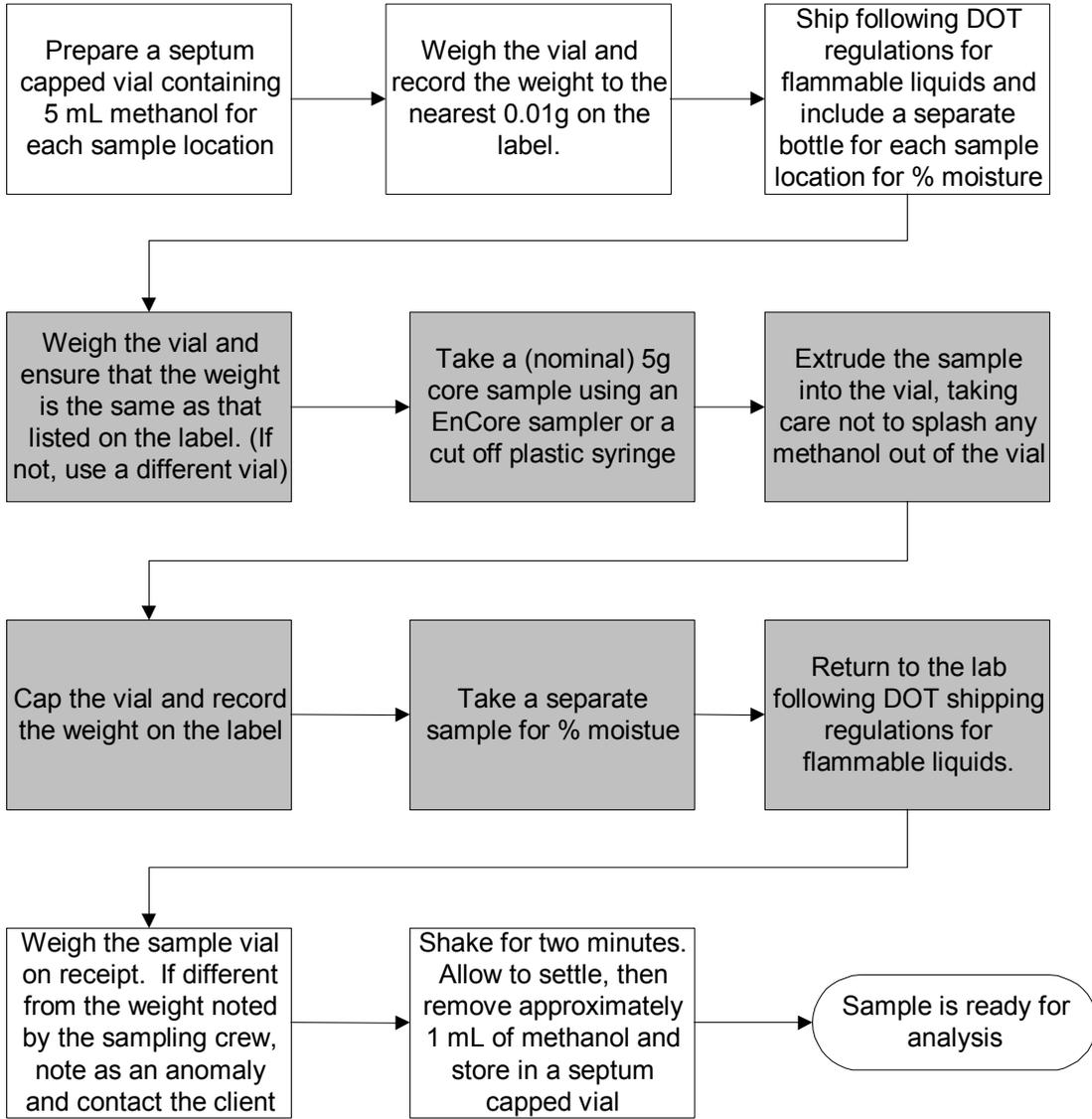
**EnCore procedure when low level is not required (field steps in gray)**



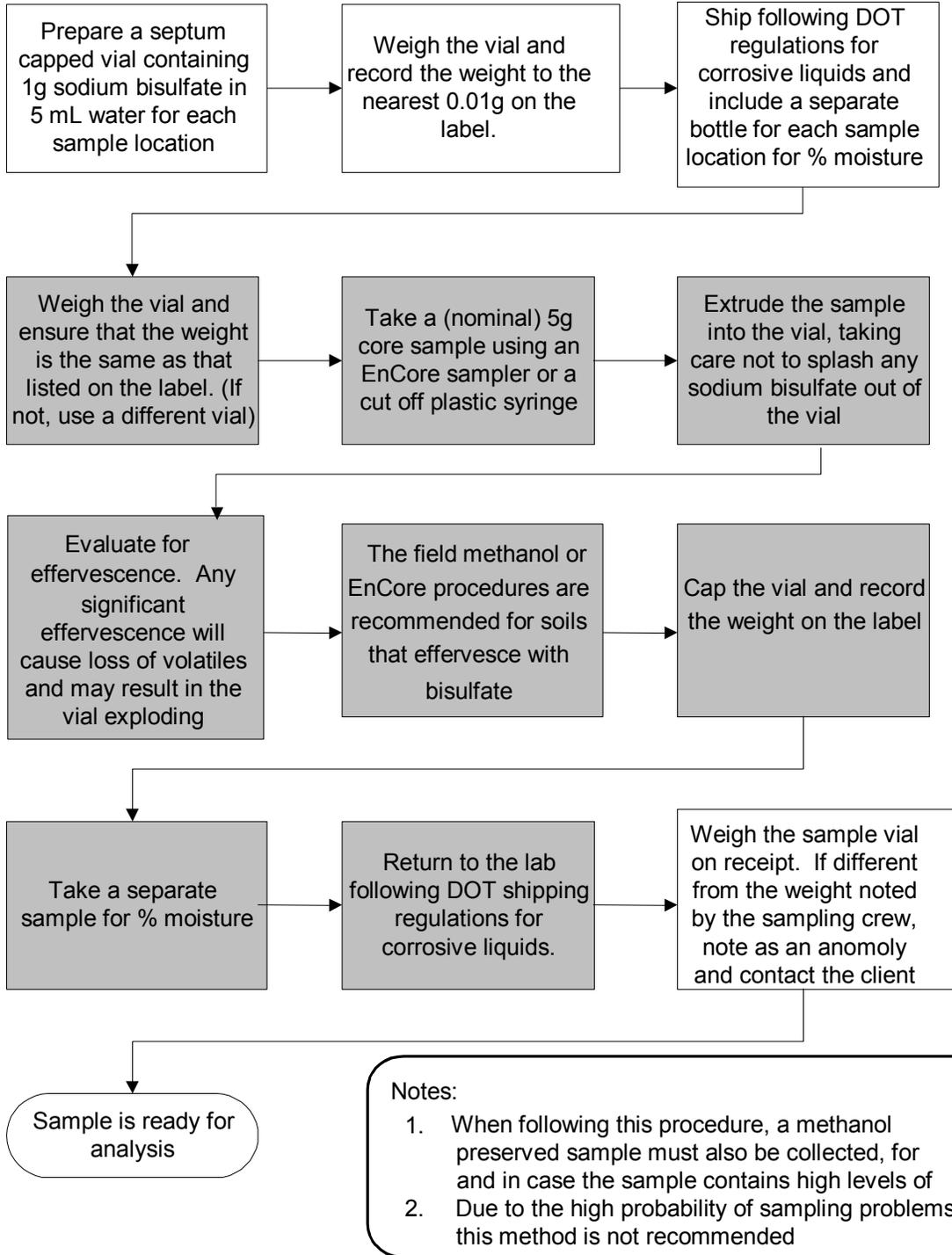
**EnCore procedure when low level is required**



**Field methanol extraction procedure (field steps in gray)**



**Field bisulfate preservation procedure (field steps in gray)**



## 9. QUALITY CONTROL

### 9.1. Control Limits

9.1.1. Control limits are established by the laboratory as described in SOP NC-QA-018.

9.1.2. Laboratory control limits are internally generated and updated periodically unless method specified. Control limits are easily accessible via LIMs (QC Browser program).

### 9.2. Surrogates

9.2.1. Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Table 8. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.
- Ensure instrument performance is acceptable.
- Recalculate the data and/or re-analyze if either of the above checks reveal a problem.
- Reprepare and re-analyze the sample if there is sufficient volume. If there is insufficient volume, the surrogate is narrated.

It is only necessary to reprepare/re-analyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out-of-control results are not due to matrix effect.

9.2.2. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and reparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then re-analysis or flagging of the data is required. For Ohio VAP samples, all surrogates must be in control, or samples must be reprepared and re-analyzed.

9.2.3. For concrete matrix, Dibromofluoromethane may have poor recovery in samples and matrix spikes. If the surrogate does not meet criteria, no further action is required due to matrix.

9.2.4. Refer to the TestAmerica QC Program document (QA-003) for further details of

the corrective actions.

### 9.3. Method Blanks

9.3.1. For each batch of samples, analyze a method blank. The method blank is analyzed after the calibration standards, normally before any samples. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of the same volume of methanol that was used to prepare the samples. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below). The method blank is acceptable if any compound detected in the blank is present in the associated samples at ten times the blank level. For Ohio VAP work, there can be no target analyte greater than the RL in the method blank unless the sample result is ND. All samples associated with an unacceptable blank will be reprepared and re-analyzed.

- 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. Such action must be taken in consultation with the client.
- Re-analysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers.

9.3.2. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples, re-extraction of the blank and affected samples will normally be required. Consultation with the client must take place. For Ohio VAP samples, all surrogates must be in control, or reparation of the batch is required.

9.3.3. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.3.4. Refer to the TestAmerica QC Program document, QA-003, for further details of the corrective actions.

9.3.5. Refer to SOP NC-QA-016 for further details concerning DoD Project Work.

#### 9.4. Laboratory Control Samples (LCS)

9.4.1. For each batch of samples, analyze an LCS. The LCS is analyzed after the calibration standard, and normally before any samples. The LCS contains a representative subset of the analytes of interest (see Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be re-preparation and re-analysis of the batch. For Ohio VAP samples, all surrogates must be in control on the LCS, or re-preparation and re-analysis of the batch is required.

- If the batch is not re-extracted and re-analyzed, the reasons for accepting the batch must be clearly presented in the project records and the report.
- If re-extraction and re-analysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.4.2. Refer to the TestAmerica QC Program document (QA-003) for further details of the corrective action.

9.4.3. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements must be negotiated with the client. n-Hexane must be spiked and reported for the LCS for Ohio VAP samples.

9.4.4. If full analyte spike lists are used at the client request, it is possible some compounds in the LCS may interfere with each other. In that case, the lab will quantitate those compounds in the LCS with a secondary ion which is free from interferences.

#### 9.5. Matrix Spikes

9.5.1. For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory-specific, historically-generated limits.

9.5.2. If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include re-analysis of the batch.
- If an MS/MSD is not possible due to limited sample, then an LCS duplicate must be analyzed. RPD of the LCS and LCSD is compared to the matrix spike limits.
- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

#### 9.6. Non-Conformance and Corrective Action

- 9.6.1. Any deviations from QC procedures must be documented as a non-conformance with applicable cause and corrective action approved by the facility QA Manager.

### 10. CALIBRATION AND STANDARDIZATION

#### 10.1. Summary

- 10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluorobenzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system.

#### 10.1.2. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	To give at least 5 scans/peak, but not to exceed 2 seconds/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 0.4 – 0.6 mL/minute

#### 10.2 Gas chromatograph suggested temperature program

##### 10.2.1 BFB Analysis

Initial Temperature: 100°C  
Initial Hold Time: 0.1 minute  
Temperature Program: 20°C/minute  
Final Temperature: 200°C

#### 10.2.2 Sample Analysis

Initial Temperature: 40°C  
Initial Hold Time: 2minutes  
Temperature Program: 15°C/minute  
Final Temperature: 200°C  
Final Hold Time: 3 minutes

### 10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each 12-hour time period. The 12-hour time period begins at the moment of injection of BFB.

### 10.4. Initial Calibration

10.4.1. A series of at least five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. Six standards must be used for a quadratic least squares calibration. Suggested calibration levels for a 5 mL purge are: 5, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Suggested calibration levels for a low level 5mL purge are 1, 5, 10, 20, and 40 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 2A, and 4 list the calibration levels for each analyte. Other calibration levels and purge volumes may be used depending on the capabilities of the specific instrument. (For example, adequate sensitivity can be obtained by using a 5 mL purge volume to reach the same reporting limits that once required a 25 mL purge. The calibration levels will still be the same 1, 5, 10, 20, 40 µg/L.) However, the same purge volume must be used for calibration and sample analysis, and the low level standard must be at or below the reporting limit.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Table 7. Target compounds must reference the nearest internal standard. Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each

compound and internal standard. See Table 13 for a list of characteristic ions. See Equation 1, Section 12, for calculation of response factor.

- 10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs. This criteria must be met before sample analysis begins.
- 10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.
- 10.4.6. Weighting of data points
- 10.4.6.1. In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason, it is preferable to increase the weighting of the lower concentration points.  $1/\text{Concentration}^2$  weighting (often called  $1/X^2$  weighting) will improve accuracy at the low end of the curve and must be used if the data system has this capability. The Y-intercept is evaluated to determine calibration acceptability.
- 10.4.7. For any analyte with % RSD >15%, linear or quadratic curve fits may be used if the compounds have historically exhibited a non-linear response. The analyst must consider instrument maintenance to improve the linearity of response. If the % RSD is > 15%, the analyst may drop the low or high in the ICAL, as long as a minimum of five points are maintained (six points for quadratic) and the quantitation range is adjusted accordingly. Otherwise, the coefficient of determination  $r^2$  must be  $\geq 0.990$ .
- 10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.
- 10.4.9. The calibration standards for the initial five-point calibration for low-level soils that are not preserved in sodium bisulfate (i.e., are preserved by freezing or not preserved) must be heated to 40°C for purging. Using this calibration curve for water samples is acceptable as long as all calibration, QC, and samples are also heated to 40°C. A separate five-point calibration must be prepared for analysis of low level soils that are preserved with sodium bisulfate. Low-level soils analysis requires the use of a closed vial autosampler such as the Varian Archon, O.I. 4552 or Tekmar Precept. Each standard for analysis of sodium bisulfate preserved

samples is prepared by spiking the methanolic standard solution through the septum of a VOA vial containing 5 mL of water and 1g sodium bisulfate. The standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging.

- 10.4.10. Non-standard analytes are sometimes requested. For these analytes, it is acceptable to analyze a single standard at the reporting limit with each continuing calibration rather than a five-point initial calibration. If the analyte is detected in any of the samples, a five-point initial calibration must be generated and the sample(s) re-analyzed for quantitation. However, if the analyte is not detected, the non-detect must be reported and no further action is necessary.

**Note:** This procedure must not be used for Ohio VAP samples.

- 10.4.11. Calibration accuracy is verified by analyzing a second source standard (ICV) immediately after the initial calibration. The recovery for CCC compounds must be  $\leq 20\%$ . The recovery for non-CCC compounds must be  $\leq 50\%$  with an allowance of up to six compounds  $> 50\%$ .

- 10.5. Continuing Calibration: The initial calibration must be verified every 12 hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. A midpoint calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in Equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be  $\leq 20\%$  for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the percent drift of all analytes must be  $\leq 50\%$  with allowance for up to six target analytes to have percent drift  $> 50\%$ .

10.5.3.1. Refer to Table 12 for specific Ohio VAP analytes.

10.5.4. If the CCCs and/or the SPCCs do not meet the criteria in Section 10.5.3 and Table 11, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample**

**quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

## 11. PROCEDURE

### 11.1. Procedural Variations

- 11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, chemistry, sample size, or other parameters. Any variation must be completely documented using a Non-Conformance Memo and approved by a Supervisor or Group Leader and QA Manager. The Non-Conformance Memo must be filed in the project file.
- 11.1.2. Any unauthorized deviations from this procedure must also be documented as a non-conformance with a cause and corrective action described. The laboratory may not deviate from the method for Ohio VAP samples.

### 11.2. Preliminary Evaluation

- 11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

### 11.3. Sample Analysis Procedure

- 11.3.1. All analysis conditions for samples must be the same as for the continuing calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).
- 11.3.2. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain an MS/MSD, an LCS, and a method blank.
  - 11.3.2.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium-level soils, the batch is defined at the sample preparation stage.
  - 11.3.2.2. It is not necessary to re-analyze batch QC with re-analyses of samples. However, any reruns must be part of a valid batch.

11.3.3 Dilutions must be done just prior to the GC/MS analysis of the sample. Dilutions are made in a Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 1  $\mu\text{L}$  of sample, then serial dilutions must be made in volumetric flasks. Dilutions may also be prepared in a 40 mL vial. An appropriate amount of water is added to the vial. The sample is added using an appropriate syringe.

11.3.3.1 The diluted concentration is to be estimated to be in the upper half of the calibration range.

#### 11.4. Methanol Extract Soils

11.4.1 Rinse a gas-tight syringe with organic free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100  $\mu\text{L}$  for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 1  $\mu\text{L}$  of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1  $\mu\text{L}$  will be added to the water in the syringe. Refer to Section 17.2 for Michigan project requirements.

#### 11.5. Liquid wastes that are soluble in methanol and insoluble in water.

11.5.1 Pipette 1 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1g.

11.5.2 Quickly add 4 mL of methanol, then add 5  $\mu\text{L}$  of a 2500  $\mu\text{g}/\text{mL}$  surrogate spiking solution to bring the final volume to 5 mL. Cap the vial and shake for two minutes to mix thoroughly. For an MS/MSD or LCS, 4.9 mL of methanol, 5  $\mu\text{L}$  of a 2500  $\mu\text{g}/\text{mL}$  surrogate spiking solution, and 0.1 mL of matrix spike solution is used.

11.5.3 Rinse a gas-tight syringe with organic-free water. Fill the syringe with the same volume of organic free water as used in the calibrations. Add no more than 2% (v/v) (100  $\mu\text{L}$  for a 5 mL purge) methanolic extract (from Sections 8.5 or 8.6) to the syringe. If less than 5  $\mu\text{L}$  of methanolic extract is to be added to the water, dilute the methanolic extract such that a volume greater than 1  $\mu\text{L}$  will be added to the water in the syringe.

#### 11.6. Aqueous and low-level soil sample analysis (Purge and Trap units that sample directly from the VOA vial)

11.6.1 Units which sample from the VOA vial must be equipped with a module which automatically adds surrogate and internal standard solution to the sample prior to

purging the sample.

11.6.2 If the autosampler uses automatic IS/SS injection, no further preparation of the VOA vial is needed. Otherwise, the internal and surrogate standards must be added to the vial. *Note:* Aqueous samples with high amounts of sediment present in the vial may not be suitable for analysis on this instrumentation, or they may need to be analyzed as soils.

11.6.3 Soil samples, which are preserved with sodium bisulfate, must be quantitated against a curve prepared with standards containing about the same amount of sodium bisulfate as the samples (1g in 5 mL).

11.6.4 Soil samples, which are preserved by freezing, must be allowed to thaw completely before sample analysis begins.

11.6.5 Sample remaining in the vial after sampling with one of these mechanisms is no longer valid for further analysis. A fresh VOA vial must be used for further sample analysis.

#### 11.7 Water Samples Not Directly Sampled from VOA Vials

11.7.1. All samples and standard solutions must be at ambient temperature before analysis.

11.7.2. Fill a syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is  $\geq 5 \mu\text{L}$ . Check and document the pH of the remaining sample.

11.7.3. Add 50 ng of each internal and surrogate standard. The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 10  $\mu\text{g/L}$  solution for a 5 mL sample). Inject the sample into the purging chamber. The internal and surrogate standards can be added automatically by the autosampler.

11.7.3.1. For TCLP samples, use 1 mL of TCLP leachate with 4 mL reagent water. (Note: TCLP reporting limits will be five times higher than the corresponding aqueous limits.)

11.7.4. Purge the sample for 11 minutes (trap must be below 35°C).

11.7.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for approximately 3-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.7.6. Desorb and bake time and temperature are optimized for the type of trap in use. The

same conditions must be used for samples and standards.

11.8. *Low-Level Solids Analysis using discrete autosamplers, Methods 8260A and 5030A*

**Note: This technique may seriously underestimate analyte concentration and must not be used except at specific client request for the purpose of comparability with previous data. It is no longer part of SW-846.**

*This method is based on purging a heated soil/sediment sample mixed with reagent water containing the surrogates and internal standards. Analyze all reagent blanks and standards under the same conditions as the samples (e.g., heated). The calibration curve is also heated during analysis. Purge temperature is 40°C.*

11.8.1. *Do not discard any supernatant liquids. Mix the contents of the container with a narrow metal spatula.*

11.8.2. *Weigh out 5g (or other appropriate aliquot) of sample into a 40 mL vial. Record the weight to the nearest 0.1g. If method sensitivity is demonstrated, a smaller aliquot may be used. Do not use aliquots less than 0.5g. If the sample is contaminated with analytes such that a purge amount less than 0.5g is appropriate, use the medium level method. For the medium level method, add 5g soil to 5 mL methanol containing the surrogates, mix for two minutes, allow to settle, then remove a portion of the methanol, and store in a clean Teflon®-capped vial at 4 °C until analysis. Analyze as described in Section 11.5.*

11.8.3. *Add 5 mL of organic free water to the VOA vial. Add surrogate/internal standard (and matrix spike solutions if required.). Add directly to the sample from Section 11.5.1.*

11.8.4. *The above steps must be performed rapidly and without interruption to avoid loss of volatile organics.*

11.9 Medium-Level Soil/Sediment and Waste Samples

11.9.1. Sediments/soils and waste that are insoluble in methanol.

11.9.1.1 Weigh 5 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.9.1.2 Quickly add 5 mL of methanol, and 5µL of 2500 µg/mL surrogate spiking solution to bring the final volume of methanol to 5 mL. For an LCS or MS/MSD sample, add 4.9 mL of methanol, 5µL of surrogate spike solution, and 0.1 mL of matrix spike solution. Cap the vial and shake or vortex to mix thoroughly.

**Note:** Sections 11.9.1.1 and 11.9.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

#### 11.10. Initial review and corrective actions

- 11.10.1. If the retention time for any internal standard in the continuing calibration changes by more than 0.5 minutes from the mid-level initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required.
- 11.10.2. If the internal standard response in the continuing calibration is more than 200% or less than 50% of the response in the mid-level of the initial calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Re-analysis of samples analyzed while the system was malfunctioning is required. Re-analysis must be undiluted if matrix interference is not observed.
  - 11.10.2.1. Any samples that do not meet the internal standard criteria for the continuing calibration must be evaluated for validity. If the change in sensitivity is a matrix effect, the sample is re-analyzed to confirm. If the change in sensitivity is due to instrumental problems, all affected samples must be re-analyzed after the problem is corrected. For Ohio VAP projects, the laboratory will re-analyze any sample where the internal standard fails, and there is no evidence of matrix interference. If there is no matrix interference, the sample must be reanalyzed at the original dilution. If the internal standard is within criteria, report the second analysis. If the internal standard is still outside of criteria, the sample must be analyzed at a second dilution. If the internal standard still does not meet criteria, the sample must be diluted until the internal standard meets criteria. Multiple runs may be required..

#### 11.11. Dilutions

- 11.11.1 If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution must be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be re-analyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.
- 11.11.2 Guidance for Dilutions Due to Matrix
  - 11.11.2.1 If the sample is initially run at a dilution and the baseline rise is less

than half the height of the internal standards, or if individual non target peaks are less than twice the height of the internal standards, then the sample must be re-analyzed at a more concentrated dilution. This requirement is approximate and subject to analyst judgement.

### 11.11.3 Reporting Dilutions

11.11.3.1 The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

## 12. DATA ANALYSIS AND CALCULATIONS

### 12.1. Qualitative identification

12.1.1 An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component, and (2) correspondence of the sample component and the standard component characteristic ions. See Table 13 for a list of the characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to coelution is evaluated.)

- The sample component retention time must compare to within  $\pm 0.2$  min. of the retention time of the standard component. For reference, the standard must be run within the same 12 hours as the sample.
- The relative intensities of ions must agree to within  $\pm 30\%$  between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.2 If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst must report that identification and proceed with quantitation.

### 12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions  $> 10\%$  of

the most abundant ion) must be present in the sample spectrum.

- 12.2.1.2. The relative intensities of the major ions must agree to within 20%.  
(Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).
- 12.2.1.3. Molecular ions present in the reference spectrum must be present in the sample spectrum.
- 12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum must be reviewed for possible background contamination or presence of coeluting compounds.
- 12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum must be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)
- 12.2.1.6. Computer-generated library search routines must not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches must the analyst assign a tentative identification.

### 12.3. Calculations

#### 12.3.1. Response factor (RF)

##### **Equation 1**

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

$A_x$  = Area of the characteristic ion for the compound to be measured

$A_{is}$  = Area of the characteristic ion for the specific internal standard

$C_{is}$  = Concentration of the specific internal standard, ng

$C_x$  = Concentration of the compound being measured, ng

### 12.3.2. Standard deviation (SD)

#### **Equation 2**

$$SD = \sqrt{\sum_{i=1}^N \frac{(X_i - X)^2}{N - 1}}$$

Where:

$X_i$  = Value of X at i through N

$N$  = Number of points

$X$  = Average value of  $X_i$

### 12.3.3. Percent relative standard deviation (%RSD):

#### **Equation 3**

$$\%RSD = \frac{\text{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$  = Mean of RF values in the curve

### 12.3.4. Percent drift between the initial calibration and the continuing calibration:

#### **Equation 4**

$$\% \text{ Drift} = \frac{C_{\text{expecte}} - C_{\text{found}}}{C_{\text{expecte}}} \times 100$$

Where:

$C_{\text{expecte}}$  = Known concentration in standard

$C_{\text{found}}$  = Measured concentration using selected quantitation method

### 12.3.5. Target compound and surrogate concentrations:

12.3.5.1 Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is  $\leq 15\%$ .

12.3.5.2 Calculation of concentration using Average Response Factors:

**Equation 5**

$$\text{Concentration } \mu\text{g} / \text{L} = \frac{x}{RF}$$

12.3.5.3 Calculation of concentration using Linear fit:

**Equation 6**

$$\text{Concentration } \mu\text{g} / \text{L} = A + Bx$$

12.3.5.4. Calculation of concentration using Quadratic fit:

**Equation 7**

$$\text{Concentration } \mu\text{g} / \text{L} = A + Bx + Cx^2$$

Where,

**x** is defined in Equations 8, 9, and 10

**A** is a constant defined by the intercept

**B** is the slope of the curve

**C** is the curvature

12.3.5.5. Calculation of **x** for Water and water-miscible waste:

**Equation 8**

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

$$X = \mu\text{g/L}$$

$A_x$  = Area of characteristic ion for the compound being measured  
(secondary ion quantitation is allowed only when there are  
sample interferences with the primary ion)

$A_{is}$  = Area of the characteristic ion for the internal standard

$I_s$  = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$V_o$  = Volume of water purged, mL

12.3.5.6. Calculation of  $x$  for Medium level soils:

**Equation 9**

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

$$X = \mu\text{g/kg}$$

$A_x$ ,  $I_s$ ,  $D_f$ ,  $A_{is}$ , same as for water.

$V_t$  = Volume of total extract, mL

$V_a$  = Volume of extract added for purging,  $\mu\text{L}$

$W_s$  = Weight of sample extracted, g

$$D = \frac{100 - \% \text{moisture}}{100}$$

12.3.5.7. Calculation of  $x$  for Low level soils:

**Equation 10**

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

$X$  = ug/kg

$A_x$ ,  $I_s$ ,  $A_{is}$ , same as for water.

$D$  is as for medium level soils

$W_s$  = Weight of sample added to the purge vessel, g

12.3.5.8. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

$A_x$  = Area in the total ion chromatogram for the compound being measured

$A_{is}$  = Area of the total ion chromatogram for the nearest internal standard without interference

$RF = 1$

In other words, the concentration is equal to  $x$  as defined in Equations 8, 9, and 10.

#### 12.3.6. MS/MSD Recovery

##### **Equation 11**

$$\text{Matrix Spike } y, \% = \frac{SSR - SR}{SA} \times 100$$

Where,

**SSR** = Spike    ple result

**SR** = Sample    sult

**SA** = Spike    ed

#### 12.3.7. Relative % Difference calculation for the MS/MSD:

##### **Equation 12**

$$RPD = \frac{|\text{MSR} - \text{MSDR}|}{\frac{1}{2}(\text{MSR} + \text{MSDR})} \times 100$$

Where:

**RPD** = Relative percent difference

**MSR** = Matrix spike result

**MSDR** = Matrix spike duplicate result

12.4 Additional equations and calculations are listed in the following SOPs: Calibration Curves (General), CA-Q-S-005, and Selection of Calibration Points, CA-T-P-002.

### **13. METHOD PERFORMANCE**

#### 13.1. Method Detection Limit

13.1.1. Generally, each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy CA-Q-S-006 and SOP NC-QA-021. When non-standard compounds are analyzed at client request, lesser requirements are possible with client agreement. At a minimum, a standard at the reporting limit must be analyzed to demonstrate the capability of the method. The non-standard compound must be detected in the reporting limit standard to be acceptable.

13.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of a standard at the reporting limit and a single point calibration.

13.2. Initial Demonstration

13.2.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.3. Training Qualification

13.3.1. The Group/Team Leader 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.

13.3.2. Method validation information (where applicable) in the form of laboratory demonstrations of capabilities is maintained for this method in the laboratory QA files.

## 14. POLLUTION PREVENTION

14.1. 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".

## 15. WASTE MANAGEMENT

15.1. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of TestAmerica. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

15.2. 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 Environmental Health and Safety Manual (CW-E-M-001) for "Waste Management and Pollution Prevention".

15.3. The following waste streams are produced when this method is carried out.

15.3.1. **Acidic material from the auto-sampler:** Waste stream must be collected and

neutralized before discharge to a sewer system if the pH is less than 5.

15.3.2. **Methanol waste from rinses and standards:** Methanol waste is discarded as a flammable liquid in a solvent waste container identified as “Flammable Liquid Waste”.

15.3.3. **All samples including purged and extracted soils and waters:** Samples are collected in boxes and removed from the lab to storage. The Waste Coordinator handles crushing the vials and proper disposal.

15.3.4. **Solid samples** - Stirbars are removed from the sample. The contents of the vial are poured into a beaker, and the soil allowed to settle out. The soil is disposed of in the solid waste container.

## 16. REFERENCES

### 16.1. References

16.1.1. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260B, Update III, December 1996

16.1.2. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994

16.1.3. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030B, Rev 2, December 1996

16.1.4. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Purge-and-Trap for Aqueous Samples, Method 5030A, Rev 1, July 1992

16.1.5. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035, Rev 0, December 1996

16.1.6. SW846, *Test Methods for Evaluating Solid Waste* Closed System Purge-and-Trap and Extraction for Volatile Organics in Soil and Waste Samples, Method 5035A, Draft Revision 1, July 2002

16.1.7. TestAmerica North Canton Quality Assurance Manual (QAM), current version

16.1.8 TestAmerica Corporate Environmental Health and Safety Manual, CW-E-M-001, and TestAmerica North Canton Facility Addendum and Contingency Plan, current version

16.2. Associated SOPs and Policies, latest version

16.2.1. QA Policy, QA-003

16.2.2. Glassware Washing, NC-QA-014

16.2.3. Statistical Evaluation of Data and Development of Control Charts, NC-QA-018

16.2.4. Method Detection Limits and Instrument Detection Limits, CA-Q-S-006 and NC-QA-021

16.2.5. Supplemental Practices for DoD Project Work, NC-QA-016

16.2.6. Standards and Reagents, NC-QA-017

16.2.7. Laboratory Holding Blanks, NC-QA-020

16.2.8. Selection of Calibration Points, CA-T-P-002

16.2.9. Calibration Curves (General), CA-Q-S-005

16.2.10. Acceptable Manual Integration Practices, CA-Q-S-002

16.2.11. Revision History

Historical File:	Revision 2.0: 12/15/97	Revision 2.4: 09/27/04
(formerly CORP-MS-0002NC)	Revision 2.1: 03/06/00	Revision 2.5: 04/03/07
	Revision 2.2: 11/28/00	Revision 0: 06/30/08 (NC-MS-019)
	Revision 2.3: 05/23/01	

**17. MISCELLANEOUS**

17.1. Modifications from the reference method

17.1.1. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

- 17.1.2. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW846 in order to improve the reliability of qualitative identification.
- 17.2 The following are protocols that must be followed to achieve the lower reporting limits required when analyzing Michigan projects.
- 17.2.1 Modify Sections 8.5.4 and 8.6.8 (add 5 uL of 2500 ug/mL surrogate solution for a nominal 10g sample).
- 17.2.2 Modify Sections 8.5.5 and 8.6.9 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).
- 17.2.3 Modify Sections 8.5.6 and 8.6.10 (add 100 uL of 50 ug/mL spike solution for a nominal 10g sample).
- 17.2.4 Michigan reporting limits for methanol preserved soils are achieved by injecting 100 uL of the methanol extract in a 5 mL purge. The instrument is calibrated using the recommended calibration range for water of 0.5 ug/L to 100 ug/L. Some analytes are prepared at higher concentrations.

**Table 1 - TestAmerica Primary Standard and Reporting Limits**

Compound	CAS Number	Reporting Limits <sup>1</sup>				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Dichlorodifluoromethane	75-71-8	5	1	5	250	250
Chloromethane	74-87-3	5	1	5	250	250
Bromomethane	74-83-9	5	1	5	250	250
Vinyl chloride	75-01-4	5	1	5	250	250
Chloroethane	75-00-3	5	1	5	250	250
Trichlorofluoromethane	75-69-4	5	1	5	250	250
Acrolein	107-02-8	100	20	100	5,000	5,000
Acetone	67-64-1	20	10	20	1,000	1,000
Trichlorotrifluoroethane	76-13-1	5	1	5	250	250
Iodomethane	74-88-4	5	1	5	250	250
Carbon disulfide	75-15-0	5	1	5	250	250
Methylene chloride	75-09-2	5	1	5	250	250
tert-Butyl alcohol	75-65-0	200	50	200	10,000	10,000
1,1-Dichloroethene	75-35-4	5	1	5	250	250
1,1-Dichloroethane	75-34-3	5	1	5	250	250
Trans-1,2-Dichloroethene	156-60-5	5.0	1.0	5.0	250	250
Acrylonitrile	107-13-1	100	20	100	5,000	5,000
Methyl tert-butyl ether (MTBE)	1634-04-4	20	5	20	1,000	1,000
Hexane	110-54-3	5	1	5	250	250
cis-1,2-Dichloroethene	156-59-2	5	1	5	250	250
1,2-Dichloroethene (Total)	540-59-0	10	2	10	500	500
Tetrahydrofuran	109-99-9	20	5	20	1,000	1,000
Chloroform	67-66-3	5	1	5	250	250
1,2-Dichloroethane	107-06-2	5	1	5	250	250
Dibromomethane	74-95-3	5	1	5	250	250
2-Butanone	78-93-3	20	5	20	1,000	1,000
1,4-Dioxane	123-91-1	500	200	500	25,000	25,000
1,1,1-Trichloroethane	71-55-6	5	1	5	250	250
Carbon tetrachloride	56-23-5	5	1	5	250	250
Bromodichloromethane	75-27-4	5	1	5	250	250
1,2-Dichloropropane	78-87-5	5	1	5	250	250
cis-1,3-Dichloropropene	10061-01-5	5	1	5	250	250
Trichloroethene	79-01-6	5	1	5	250	250
Dibromochloromethane	124-48-1	5	1	5	250	250
1,2-Dibromoethane	106-93-4	5	1	5	250	250

**Table 1 - TestAmerica Primary Standard and Reporting Limits**

Compound	CAS Number	Reporting Limits <sup>1</sup>				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
1,2,3-Trichloropropane	96-18-4	5	1	5	250	250
1,1,2-Trichloroethane	79-00-5	5	1	5	250	250
Benzene	71-43-2	5	1	5	250	250
Ethylmethacrylate	97-63-2	5	1	5	250	250
Trans-1,3-Dichloropropene	10061-02-6	5	1	5	250	250
Bromoform	75-25-2	5	1	5	250	250
4-Methyl-2-pentanone	108-10-1	20	5	20	1000	1,000
2-Hexanone	591-78-6	20	5	20	1000	1,000
Tetrachloroethene	127-18-4	5	1	5	250	250
Toluene	108-88-3	5	1	5	250	250
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	250	250
2-Chloroethyl vinyl ether	110-75-8	N/A <sup>2</sup>	N/A	50	1000	1,000
Vinyl acetate	108-05-4	10	2	10	500	500
Chlorobenzene	108-90-7	5	1	5	250	250
Ethylbenzene	100-41-4	5	1	5	250	250
Styrene	100-42-5	5	1	5	250	250
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	250	250
m and p Xylenes		10	2	10	500	500
o-xylene	95-47-6	5.0	1	5	250	250
Total xylenes	1330-20-7	10	2	10	500	500
1,3-Dichlorobenzene	541-73-1	5	1	5	250	250
1,4-Dichlorobenzene	106-46-7	5	1	5	250	250
1,2-Dichlorobenzene	95-50-1	5	1	5	250	250
2,2-Dichloropropane	590-20-7	5	1	5	250	
Bromochloromethane	74-97-5	5	1	5	250	
1,1-Dichloropropene	563-58-6	5	1	5	250	
Bromodichloromethane	75-27-4	5	1	5	250	
1,2-Dichloropropane	78-87-5	5	1	5	250	
1,3-Dichloropropane	142-28-9	5	1	5	250	
Isopropylbenzene	98-82-8	5	1	5	250	
Bromobenzene	108-86-1	5	1	5	250	
n-Propylbenzene	103-65-1	5	1	5	250	
2-Chlorotoluene	95-49-8	5	1	5	250	
4-Chlorotoluene	106-43-4	5	1	5	250	
1,3,5-Trimethylbenzene	108-67-8	5	1	5	250	

**Table 1 - TestAmerica Primary Standard and Reporting Limits**

Compound	CAS Number	Reporting Limits <sup>1</sup>				
		5 mL Water µg/L	Low Level 5 mL water µg/L	Low soil µg/kg	8260B/ 5035 Soil ug/kg	8260A 5030A Med Level Soil µg/kg
Tert-Butylbenzene	98-06-6	5	1	5	250	
1,2,4-Trimethylbenzene	95-63-6	5	1	5	250	
Sec-butylbenzene	135-98-8	5	1	5	250	
4-Isopropyltoluene	99-87-6	5	1	5	250	
n-Butylbenzene	104-51-8	5	1	5	250	
1,2,4-Trichlorobenzene	120-82-1	5	1	5	250	
Napthalene	91-20-3	5	1	5	250	
Hexachlorobutadiene	87-68-3	5	1	5	250	
1,2,3-Trichlorobenzene	87-61-6	5	1	5	250	
Acetonitrile	75-05-8	100	20	100	5000	
Cyclohexane	110-82-7	10	1	10	500	
Methyl Acetate	79-20-9	10	10	10	500	
Methyl cyclohexane	108-87-2	10	1	10	500	

<sup>1</sup> Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment, calculated on dry weight basis, will be higher.

<sup>2</sup> 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

**Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	5	20	50	100	200
Toluene-d8 (Surrogate)	5	20	50	100	200
4-Bromofluorobenzene (Surrogate)	5	20	50	100	200
Dichlorodifluoromethane	5	20	50	100	200
Chloromethane	5	20	50	100	200
Bromomethane	5	20	50	100	200
Vinyl chloride	5	20	50	100	200
Chloroethane	5	20	50	100	200
Trichlorofluoromethane	5	20	50	100	200
Acrolein	50	200	500	1000	2000
Acetone	5	20	50	100	200
Trichlorotrifluoroethane	5	20	50	100	200
Iodomethane	5	20	50	100	200
Carbon disulfide	5	20	50	100	200
Methylene chloride	5	20	50	100	200
tert-Butyl alcohol	100	400	1,000	2,000	4,000
1,1-Dichloroethene	5	20	50	100	200
1,1-Dichloroethane	5	20	50	100	200
trans-1,2-Dichloroethene	5	20	50	100	200
Acrylonitrile	50	200	500	1,000	2,000
Methyl tert-butyl ether (MTBE)	5	20	50	100	200
Hexane	5	20	50	100	200
cis-1,2-Dichloroethene	5	20	50	100	200
Tetrahydrofuran	5	20	50	100	200
Chloroform	5	20	50	100	200
1,2-Dichloroethane	5	20	50	100	200
Dibromomethane	5	20	50	100	200
2-Butanone	5	20	50	100	200
1,4-Dioxane	250	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	5	20	50	100	200
Carbon tetrachloride	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
cis-1,3-Dichloropropene	5	20	50	100	200
Trichloroethene	5	20	50	100	200
Dibromochloromethane	5	20	50	100	200
1,2-Dibromoethane	5	20	50	100	200
1,2,3-Trichloropropane	5	20	50	100	200
Acetonitrile	50	200	500	1000	2000

**Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,1,2-Trichloroethane	5	20	50	100	200
Benzene	5	20	50	100	200
Ethylmethacrylate	5	20	50	100	200
trans-1,3-Dichloropropene	5	20	50	100	200
Bromoform	5	20	50	100	200
4-Methyl-2-pentanone	5	20	50	100	200
2-Hexanone	5	20	50	100	200
Tetrachloroethene	5	20	50	100	200
Toluene	5	20	50	100	200
1,1,2,2-Tetrachloroethane	5	20	50	100	200
2-Chloroethyl vinyl ether	10	40	100	200	400
Vinyl acetate	5	20	50	100	200
Chlorobenzene	5	20	50	100	200
Ethylbenzene	5	20	50	100	200
Styrene	5	20	50	100	200
t-1,4-Dichloro-2-butene	5	20	50	100	200
m and p Xylenes	10	40	100	200	400
o-xylene	5	20	50	100	200
1,3-Dichlorobenzene	5	20	50	100	200
1,4-Dichlorobenzene	5	20	50	100	200
1,2-Dichlorobenzene	5	20	50	100	200
2,2-Dichloropropane	5	20	50	100	200
Bromochloromethane	5	20	50	100	200
1,1-Dichloropropene	5	20	50	100	200
Bromodichloromethane	5	20	50	100	200
1,2-Dichloropropane	5	20	50	100	200
1,3-Dichloropropane	5	20	50	100	200
Isopropylbenzene	5	20	50	100	200
Bromobenzene	5	20	50	100	200
n-Propylbenzene	5	20	50	100	200
2-Chlorotoluene	5	20	50	100	200
4-Chlorotoluene	5	20	50	100	200
1,3,5-Trimethylbenzene	5	20	50	100	200
tert-Butylbenzene	5	20	50	100	200
1,2,4-Trimethylbenzene	5	20	50	100	200
sec-butylbenzene	5	20	50	100	200
4-Isopropyltoluene	5	20	50	100	200
n-Butylbenzene	5	20	50	100	200
1,2,4-Trichlorobenzene	5	20	50	100	200
Napthalene	5	20	50	100	200

**Table 2 - TestAmerica Primary Standard Calibration Levels, 5 mL purge**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Hexachlorobutadiene	5	20	50	100	200
1,2,3-Trichlorobenzene	5	20	50	100	200

**Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level <sup>1</sup>**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Dibromofluoromethane (Surrogate)	1	5	10	20	40
1,2-Dichloroethane-d4 (Surrogate)	1	5	10	20	40
Toluene-d8 (Surrogate)	1	5	10	20	40
Bromofluorobenzene (Surrogate)	1	5	10	20	40
Dichlorodifluoromethane	1	5	10	20	40
Chloromethane	1	5	10	20	40
Vinyl Chloride	1	5	10	20	40
Bromomethane	1	5	10	20	40
Chloroethane	1	5	10	20	40
Trichlorofluoromethane	1	5	10	20	40
Acrolein	10	50	100	200	400
Acetone	2	10	20	40	80
1,1-Dichloroethene	1	5	10	20	40
Trichlorotrifluoroethane	1	5	10	20	40
Iodomethane	1	5	10	20	40
Carbon Disulfide	1	5	10	20	40
Methylene Chloride	1	5	10	20	40
Acetonitrile	10	50	100	200	400
Acrylonitrile	10	50	100	200	400
Methyl tert-butyl ether	1	5	10	20	40
trans-1,2-Dichloroethene	1	5	10	20	40
Hexane	1	5	10	20	40
Vinyl acetate	1	5	10	20	40
1,1-Dichloroethane	1	5	10	20	40
tert-Butyl Alcohol	20	100	200	400	800
2-Butanone	2	10	20	40	80
cis-1,2-dichloroethene	1	5	10	20	40
2,2-Dichloropropane	1	5	10	20	40
Bromochloromethane	1	5	10	20	40
Chloroform	1	5	10	20	40

**Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level <sup>1</sup>**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Tetrahydrofuran	1	5	10	20	40
1,1,1-Trichloroethane	1	5	10	20	40
1,1-Dichloropropene	1	5	10	20	40
Carbon Tetrachloride	1	5	10	20	40
1,2-Dichloroethane	1	5	10	20	40
Benzene	1	5	10	20	40
Trichloroethene	1	5	10	20	40
1,2-Dichloropropane	1	5	10	20	40
1,4-Dioxane	50	250	500	1000	2000
Dibromomethane	1	5	10	20	40
Bromodichloromethane	1	5	10	20	40
2-Chloroethyl vinyl ether	2	10	20	40	80
cis-1,3-Dichloropropene	1	5	10	20	40
4-Methyl-2-pentanone	2	10	20	40	80
Toluene	1	5	10	20	40
trans-1,3-Dichloropropene	1	5	10	20	40
Ethyl Methacrylate	1	5	10	20	40
1,1,2-Trichloroethane	1	5	10	20	40
1,3-Dichloropropane	1	5	10	20	40
Tetrachloroethene	1	5	10	20	40
2-Hexanone	2	10	20	40	80
Dibromochloromethane	1	5	10	20	40
1,2-Dibromoethane	1	5	10	20	40
Chlorobenzene	1	5	10	20	40
1,1,1,2-Tetrachloroethane	1	5	10	20	40
Ethylbenzene	1	5	10	20	40
m + p-Xylene	2	10	20	40	80
Xylene-o	1	5	10	20	40
Styrene	1	5	10	20	40
Bromoform	1	5	10	20	40
Isopropylbenzene	1	5	10	20	40
1,1,2,2-Tetrachloroethane	1	5	10	20	40
1,4-Dichloro-2-butene	1	5	10	20	40
1,2,3-Trichloropropane	1	5	10	20	40
Bromobenzene	1	5	10	20	40
n-Propylbenzene	1	5	10	20	40
2-Chlorotoluene	1	5	10	20	40
1,3,5-Trimethylbenzene	1	5	10	20	40
4-Chlorotoluene	1	5	10	20	40

**Table 2A - TestAmerica Primary Standard Calibration Levels, Low Level <sup>1</sup>**

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
tert-Butylbenzene	1	5	10	20	40
1,2,4-Trimethylbenzene	1	5	10	20	40
sec-Butylbenzene	1	5	10	20	40
4-Isopropyltoluene	1	5	10	20	40
1,3-Dichlorobenzene	1	5	10	20	40
1,4-Dichlorobenzene	1	5	10	20	40
n-Butylbenzene	1	5	10	20	40
1,2-Dichlorobenzene	1	5	10	20	40
1,2-Dibromo-3-chloropropane	1	5	10	20	40
1,2,4-Trichlorobenzene	1	5	10	20	40
Hexachlorobutadiene	1	5	10	20	40
Naphthalene	1	5	10	20	40
1,2,3-Trichlorobenzene	1	5	10	20	40
Cyclohexane	1	5	10	20	40
Methyl Acetate	2	10	20	40	80
Methylcyclohexane	1	5	10	20	40
1,3,5-Trichlorobenzene	1	5	10	20	40

<sup>1</sup> 25 mL purge samples analyzed at 5 mL purge on more sensitive equipment.

**Table 3 - TestAmerica Appendix IX Standard and Reporting Limits, 5 mL purge**

Compound	CAS Number	Reporting Limits			
		5 mL Water $\mu\text{g/L}$	Low Level 5mL purge water $\mu\text{g/L}$	Low Soil $\mu\text{g/kg}$	Medium Soil $\mu\text{g/mL}$
Allyl Chloride	107-05-1	10	2	10	500
Dichlorofluoromethane	75-43-4	10	2	10	500
Isopropyl ether	108-20-3	10	2	10	500
Chloroprene	126-99-8	5	2	5	250
n-Butanol	71-36-3	200	50	200	10,000
Propionitrile	107-12-0	20	4	20	1000
Methacrylonitrile	126-98-7	5	2	5	250
Isobutanol	78-83-1	200	50	200	10,000
Methyl methacrylate	80-62-6	5	2	5	250
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	250
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	500
Ethyl ether	60-29-7	10	2	10	500
Ethyl Acetate	141-78-6	20	4	20	1,000
2-Nitropropane	79-46-9	10	4	10	500
Cyclohexanone	108-94-1	50	20	50	2500
Isopropylbenzene	98-82-8	5	1	5	250
2-Methylnaphthalene (Michigan only)	91-57-6	NA	5	NA	330

**Table 4**  
**Recommended/TestAmerica Appendix IX Standard Calibration Levels, µg/L**

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	5	20	50	100	200
Dichlorofluoromethane	5	20	50	100	200
Isopropyl ether	5	20	50	100	200
Chloroprene	5	20	50	100	200
n-Butanol	100	400	1,000	2,000	4,000
Propionitrile	10	40	100	200	400
Methacrylonitrile	5	20	50	100	200
Isobutanol	100	400	1,000	2,000	4,000
Methyl methacrylate	5	20	50	100	200
1,1,1,2-Tetrachloroethane	5	20	50	100	200
1,2-Dibromo-3-chloropropane	10	40	100	200	400
Ethyl ether	5	20	50	100	200
Ethyl Acetate	10	40	100	200	400
2-Nitropropane	10	40	100	200	400
Cyclohexanone	50	200	500	1,000	2,000
2-Methylnaphthalene (Michigan only)	2	10	20	40	80
Ethyl tert-butyl ether	5	20	50	100	200
tert-Amyl methyl ether	5	20	50	100	200
1,2,3-Trimethylbenzene	5	20	50	100	200

**Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard**

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
Dichlorodifluoromethane	75-71-8			X	X
Chloromethane	74-87-3	X		X	X
Bromomethane	74-83-9	X		X	X
Vinyl chloride	75-01-4	X	X	X	X
Chloroethane	75-00-3	X		X	X
Trichlorofluoromethane	75-69-4			X	X
Acrolein	107-02-8				X
Acetone	67-64-1	X		X	X
Trichlorotrifluoroethane	76-13-1				
Iodomethane	74-88-4				X
Carbon disulfide	75-15-0	X		X	X
Methylene chloride	75-09-2	X		X	X
tert-Butyl alcohol	75-65-0				
1,1-Dichloroethene	75-35-4	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X
Total 1,2-Dichloroethene		X		X	X
Acrylonitrile	107-13-1				X
Methyl <i>tert</i> -butyl ether (MTBE)	1634-04-4			X	
Hexane	110-54-3				
cis-1,2-Dichloroethene	156-59-2	X		X	
Tetrahydrofuran	109-99-9				
Chloroform	67-66-3	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X
Dibromomethane	74-95-3				X
2-Butanone	78-93-3	X	X	X	X
1,4-Dioxane	123-91-1				X
1,1,1-Trichloroethane	71-55-6	X		X	X
Carbon tetrachloride	56-23-5	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X
1,2-Dichloropropane	78-87-5	X		X	X
cis-1,3-Dichloropropene	10061-01-5	X		X	X
Trichloroethene	79-01-6	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X
1,2-Dibromoethane	106-93-4			X	X
1,2,3-Trichloropropane	96-18-4				X
1,1,2-Trichloroethane	79-00-5	X		X	X

**Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard**

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
Benzene	71-43-2	X	X	X	X
Ethylmethacrylate	97-63-2				X
trans-1,3-Dichloropropene	10061-02-6	X		X	X
Bromoform	75-25-2	X		X	X
4-Methyl-2-pentanone	108-10-1	X		X	X
2-Hexanone	591-78-6	X		X	X
Tetrachloroethene	127-18-4	X	X	X	X
Toluene	108-88-3	X		X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X
2-Chloroethyl vinyl ether	110-75-8				
Vinyl acetate	108-05-4				X
Chlorobenzene	108-90-7	X	X	X	X
Ethylbenzene	100-41-4	X		X	X
Styrene	100-42-5	X		X	X
t-1,4-Dichloro-2-butene	110-57-6				X
m and p Xylenes		X		X	X
o-xylene	95-47-6	X		X	X
Total xylenes	1330-20-7	X		X	X
1,3-Dichlorobenzene	541-73-1	X		X	
1,4-Dichlorobenzene	106-46-7	X		X	
1,2-Dichlorobenzene	95-50-1	X		X	
Cyclohexane	110-82-7	X		X	
Methyl Acetate	79-20-9	X		X	
Methyl cyclohexane	108-87-2	X		X	
Isopropylbenzene	98-82-8			X	
1,2-Dibromo-3-chloropropane	96-12-8			X	X
1,2,4-Trichlorobenzene	120-82-1			X	
Acetonitrile	75-05-8				X
1,1,1,2-Tetrachloroethane	630-20-6				X
2,2-Dichloropropene	594-20-7				
Bromochloromethane	74-97-5				
1,1-Dichloropropene	563-58-6				
1,3-Dichloropropane	142-28-9				
Bromobenzene	108-86-1				
n-Propylbenzene	103-65-1				
2-Chlorotoluene	95-49-8				
1,3,5-Trimethylbenzene	108-67-8				
4-Chlorotoluene	106-43-4				
tert-Butylbenzene	98-06-6				
1,2,4-Trimethylbenzene	95-63-6				

**Table 5 - Reportable Analytes for TestAmerica Standard Tests, Primary Standard**

Compound	CAS Number	TestAmerica Standard List	TCLP	TCL	Appendix IX
sec-Butylbenzene	135-98-8				
4-Isopropyltoluene	99-87-6				
n-Butylbenzene	104-51-8				
Hexachlorobutadiene	87-68-3				
Naphthalene	91-20-3				
1,2,3-Trichlorobenzene	87-61-6				
1,3,5-Trichlorobenzene	108-70-3				

**Table 6**

**Reportable Analytes for TestAmerica Standard Tests, Appendix IX standard**

Compound	Number	Appendix IX
Allyl Chloride	107-05-1	X
Dichlorofluoromethane	75-43-4	
Isopropyl ether	108-20-3	
Chloroprene	126-99-8	X
n-Butanol	71-36-3	
Propionitrile	107-12-0	X
Methacrylonitrile	126-98-7	X
Isobutanol	78-83-1	X
Methyl methacrylate	80-62-6	X
Ethyl ether	60-29-7	
Ethyl Acetate	141-78-6	
2-Nitropropane	79-46-9	
Cyclohexanone	108-94-1	
Ethyl tert-butyl ether	637-92-3	
tert-Amyl methyl ether	994-05-8	
1,2,3-trimethylbenzene	526-73-8	

**Table 7 - Internal Standards**

Compound	Standard Concentration µg/mL (may vary per matrix)	Quantitation ion (5 mL purge)
Fluorobenzene	50 – 250	96
Chlorobenzene-d5	50 – 250	117
1,4-Dichlorobenzene-d4	50 – 250	152

Notes:

- 1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

**Table 8 - Surrogate Standards**

Surrogate Compounds	Standard Concentration µg/mL (may vary per matrix)
1,2-Dichloroethane-d <sub>4</sub>	50 – 250
Dibromofluoromethane	50 – 250
Toluene-d <sub>8</sub>	50 - 250
4-Bromofluorobenzene	50 – 250

Notes:

- 1) Except for medium level soils, the surrogate and internal standards may be combined in one solution.
- 2) Recovery limits for surrogates are generated from historical data and are maintained by the QA Dept.

**Table 9 - Matrix Spike / LCS Control  
 Compounds**

Compound	Standard Concentration µg /mL
1,1,1-Trichloroethane	50 - 250
1,1,2,2-Tetrachloroethane	50
1,1,2-Trichloro-1,2,2-trifluoroethane	50
1,1,2-Trichloroethane	50
1,1-Dichloroethane	50
1,1-Dichloroethene	50
1,1-Dichloropropene	50
1,2,3-Trichlorobenzene	50
1,2,3-Trichloropropane	50
1,2,4-Trichlorobenzene	50
1,2,4-Trimethylbenzene	50
1,2-Dibromo-3-chloropropane	50
1,2-Dibromoethane	50
1,2-Dichlorobenzene	50
1,2-Dichloroethane	50
1,2-Dichloroethene (total)	100
1,2-Dichloropropane	50
1,3,5-Trimethylbenzene	50
1,3-Dichlorobenzene	50
1,3-Dichloropropane	50
1,4-Dichlorobenzene	50
2,2-Dichloropropane	50
2-Butanone	50
2-Chloroethyl Vinyl Ether	100 - 500
2-Chlorotoluene	50
2-Hexanone	50
4-Chlorotoluene	50
4-Methyl-2-pentanone	50
Acetone	50
Acetonitrile	500 – 2500
Acrolein	500
Acrylonitrile	100 - 500
Benzene	50
Bromobenzene	50
Bromochloromethane	50
Bromodichloromethane	50
Bromoform	50
Bromomethane	50
Carbon disulfide	50
Carbon tetrachloride	50
Chlorobenzene	50
Chloroethane	50

**Table 9 - Matrix Spike / LCS Control  
 Compounds**

Compound	Standard Concentration µg /mL
Chloroform	50
Chloromethane	50
cis-1,2-Dichloroethene	50
cis-1,3-Dichloropropene	50
Cyclohexane	50
Dibromochloromethane	50
Dibromomethane	50
Dichlorodifluoromethane	50
Ethylbenzene	50
Hexachlorobutadiene	50
Iodomethane	50
Isopropylbenzene	50
Isopropylether	50
Methyl acetate	50
Methyl tert-butyl ether (MTBE)	50
Methylcyclohexane	50
Methylene chloride	50
Naphthalene	50
n-Butylbenzene	50
n-Hexane (Ohio VAP only)	50
n-Propylbenzene	50
p-Isopropyltoluene	50
sec-Butylbenzene	50
Styrene	50
tert-Butylbenzene	50
Tetrachloroethene	50
Toluene	50
trans-1,2-Dichloroethene	50
trans-1,2-Dichloroethene	50
trans-1,3-Dichloropropene	50
Trichloroethene	50
Trichlorofluoromethane	50
Vinyl Acetate	50
Vinyl chloride	50
Xylenes (total)	150 - 750

- Notes: 1) 5 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.
- 2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by QA Dept.

**Table 10 - BFB Key Ion Abundance Criteria**

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

**Table 11 - SPCC Compounds and Minimum Response Factors**

Compound	8260B, 8260A Min. RF
Chloromethane	0.100
1,1-Dichloroethane	0.100
Bromoform	0.100
1,1,2,2-Tetrachloroethane	0.300
Chlorobenzene	0.300

**Table 12 - CCC compounds**

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	30	20
1,1-Dichloroethene	30	20
Chloroform	30	20
1,2-Dichloropropane	30	20
Toluene	30	20
Ethylbenzene	30	20
n-Hexane (Ohio VAP only)	30	20

**Table 13 - Characteristic Ions**

Compound	Primary*	Secondary	Tertiary
1,2-Dichloroethane-d <sub>4</sub> (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101, 103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl <i>tert</i> butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98

**Table 13 - Characteristic Ions**

Compound	Primary*	Secondary	Tertiary
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Toluene-d <sub>8</sub> (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100

**Table 13 - Characteristic Ions**

Compound	Primary*	Secondary	Tertiary
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	
Cyclohexane	56	69	84
Methyl Acetate	43	74	
Methyl cyclohexane	83	55	98

\* The primary ion must be used for quantitation unless interferences are present, in which case a secondary ion may be used.

\*\* m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

# CORPORATE QUALITY MANAGEMENT PLAN

## Analytical Laboratories

Revision: 0

January 2009

**Copyright Information:**

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

**THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:**

**©COPYRIGHT 2009 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.**

Controlled Source: **Intranet**

Facility Distribution No. \_\_\_\_\_

## Corporate Quality Management Plan

### Approval Signatures



---

Rachel Brydon Jannetta  
President & Chief Executive Officer (CEO)

1/6/2009

---

Date



---

Dr. Keith Wheatstone  
Chief Operating Officer (COO)

1/5/2009

---

Date



---

Dr. Charles W. Carter  
Vice President of Quality & Technical Services (QTS)

12/16/2008

---

Date



---

Raymond J. Frederici  
Director of Quality & Client Advocacy

12/15/2008

---

Date

## Table of Contents

Section No.	Title	Page No.
-	Cover Page	1
-	Approval Signatures	2
2.0	Table of Contents	3
3.0	Introduction	6
3.1	Overview	6
3.2	Purpose	7
3.3	References	7
3.4	Scope	8
3.5	Terms And Definitions	9
4.0	Organization and Management	9
4.1	Roles And Responsibilities	9
5.0	Quality System	14
5.1	Quality Assurance Policy	14
5.2	Management Commitment To Quality Assurance And Data Integrity	14
5.3	Objectives Of The Quality System	14
6.0	Document Control	16
6.1	Document Type	16
6.2	Document Control Procedure	17
6.3	Document Revision	17
6.4	Official Documents	17
7.0	Service to the Client	17
7.1	Contract Review	17
7.2	Project Specific Quality Planning	18
7.3	Client Confidentiality	18
7.4	Client Surveys	19
8.0	Subcontracting	19
9.0	Purchasing Services and Supplies	19
10.0	Complaints	20
11.0	Control of Non-Conforming Work	20
12.0	Corrective Action	21
12.1	General	21
12.2	Initiation	21
12.3	Cause Analysis	21
12.4	Corrective Action	21
12.5	Monitoring Corrective Action	22
13.0	Preventative Action	22
13.1	Management Of Change	22
14.0	Control of Records	23
14.1	Record Types & Record Retention	23
14.2	Programs With Longer Retention Requirements	24

<b>Section No.</b>	<b>Title</b>	<b>Page No.</b>
14.3	Archives And Record Transfer	24
15.0	Audits	25
15.1	Internal Audits - Audit Types And Frequency	25
15.2	External Audits	26
15.3	Audit Findings	27
16.0	Management Reviews	27
16.1	QA Reports To Management	27
16.2	Management Systems Review	28
17.0	Personnel	28
17.1	General	28
18.0	Accommodations & Environmental Conditions	31
19.0	Test Methods and Method Validation	31
19.1	Test Methods	31
19.2	Standard Operating Procedures	33
19.3	Method Validation And Verification Activities For All New Methods	34
19.4	Permitting Departures From Documented Procedure	35
20.0	Equipment (and Calibration)	36
20.1	Equipment Operation	36
20.2	Equipment Maintenance	36
20.3	Equipment Verification And Calibration	36
20.4	Calibration	36
20.5	Glassware Cleaning	37
20.6	Data Integrity And Security	37
21.0	Measurement Traceability	38
21.1	General	38
21.2	Reference Standards Traceability	38
21.3	Reagents	39
22.0	Sampling	39
22.1	Sampling Plans	39
23.0	Handling of Samples	39
23.1	General	39
23.2	Sample Acceptance Policy	40
23.3	Sample Identification And Traceability	40
23.4	Sub-Sampling	41
23.5	Sample Preparation	41
23.6	Sample Disposal	41
24.0	Assuring the Quality of Test Results	41
24.1	Control Samples	41
24.2	Review / Verification Procedures	42
24.3	Development Of QC Criteria, Non-Specified In Method/Regulation	44
25.0	Reporting Results	44
25.1	Project Reports	44
25.2	Project Report Content	44
25.3	Electronic Data Deliverables	46

<b>Section No.</b>	<b>Title</b>	<b>Page No.</b>
25.4	Project Report Format	46

## Tables & Figures

<b>Table No.</b>	<b>Title</b>	<b>Page No.</b>
3-1	TestAmerica Analytical Facility Locations	6
14-1	Example of TestAmerica Record Types	23
14-2	Example: Special Record Retention Requirements	24
15-1	Types of Internal Audits and Frequency	25
17-1	TestAmerica Employee Minimum Training Requirements	29
24-1	Example of Control Samples	42

<b>Figure No.</b>	<b>Title</b>	<b>Page No.</b>
4-1	TestAmerica Organizational Chart	13
17-1	Example Demonstration of Capability Certification Statement	30
19-1	Proprietary Information Statement	34

## Appendix

<b>Appendix No.</b>	<b>Title</b>	<b>Page No.</b>
1	List of TestAmerica Corporate Policies & SOPs	47

### 3.0 Introduction

#### 3.1 Overview

TestAmerica is the leading environmental testing firm in the United States, including 38 laboratories and 27 service centers. TestAmerica provides innovative technical expertise and comprehensive analytical testing services. Specialty analyses include source and ambient air, aquatic toxicity, explosives, specialty organics, dioxins, drinking water, sediments and tissues, emerging contaminants, radiochemistry and mixed waste testing. TestAmerica is well positioned to support a variety of clients including commercial, governmental and chemical industries.

TestAmerica offers a broad range of environmental testing services. TestAmerica's testing capabilities include chemical, physical, and biological analyses of a variety of matrices, including aqueous, solid, drinking water, waste, tissue, air, mold and fungus (Mycology) and saline/estuarine samples. Specialty capabilities include air toxics testing, radiological, mixed waste testing, geotechnical testing, tissue preparation and analysis, aquatic toxicology, dioxin/furan testing, indoor air quality and microscopy services, asbestos analysis, High Resolution Mass Spectrometry (HRMS), Inductively Coupled Plasma/MS (ICP/MS), Liquid Chromatography/MS (LC/MS), PCR microbiology and on-site technologies including mobile laboratories. TestAmerica laboratory locations are as follows:

**Table 3-1. TestAmerica Analytical Facility Locations**

TestAmerica Anchorage	TestAmerica North Canton
TestAmerica Austin	TestAmerica Ontario
TestAmerica Buffalo	TestAmerica Pensacola
TestAmerica Burlington	TestAmerica Phoenix
TestAmerica Cedar Falls	TestAmerica Pittsburgh
TestAmerica Chicago	TestAmerica Portland
TestAmerica Connecticut	TestAmerica Richland
TestAmerica Corpus Christi	TestAmerica San Francisco
TestAmerica Dayton	TestAmerica Savannah
TestAmerica Denver	TestAmerica Seattle
TestAmerica Edison	TestAmerica Spokane
TestAmerica Honolulu	TestAmerica St. Louis
TestAmerica Houston	TestAmerica Tacoma
TestAmerica Irvine	TestAmerica Tallahassee
TestAmerica King of Prussia	TestAmerica Tampa
TestAmerica Knoxville	TestAmerica Valparaiso
TestAmerica Los Angeles	TestAmerica Watertown
TestAmerica Mobile	TestAmerica West Sacramento
TestAmerica Nashville	TestAmerica Westfield

### 3.2 Purpose

The purpose of TestAmerica's Corporate Quality Management Plan (CQMP) is to describe the TestAmerica quality system and to outline how that system enables all employees of TestAmerica to meet the Quality Assurance (QA) Policy. This document also describes specific QA activities and requirements and prescribes their frequencies. Roles and responsibilities of TestAmerica Senior Management in support of the quality system are also defined.

### 3.3 References

The following references were used in preparation of this document and as the basis of the TestAmerica Quality System:

- ❖ National Environmental Laboratory Accreditation Conference (NELAC) Standard, 2003
- ❖ 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; Update III, December 1996; and Update IV, February 2007.
- ❖ 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.
- ❖ U.S. Department of Energy Order 414.1B, *Quality Assurance*, 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.
- ❖ U.S. Department of Defense, *Quality Systems Manual for Environmental Laboratories*, Final Version 3, January 2006.
- ❖ U.S. Department of Defense, *Air Force Center for Environmental Excellence Quality Assurance Project Plan (QAPP)*, Version 4.0.02, May 2006.
- ❖ Nuclear Regulatory Commission (NRC) quality assurance requirements.
- ❖ Marine Protection, Research, and Sanctuaries Act (MPRSA).
- ❖ Toxic Substances Control Act (TSCA).

### 3.4 Scope

The requirements set forth in this document are applicable to all TestAmerica laboratories. Where this document uses the terms "must" and "shall", this denotes required activities. Practices described in this CQMP denote how those activities are performed in general; and each laboratory may have a more detailed description of that activity.

Each laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where this CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. Each laboratory's Quality Assurance Manual (QAM) shall take precedence over the CQMP in those cases. Secondly, each TestAmerica laboratory has the responsibility and authority to operate in compliance with documented client requirements, where they do not conflict with regulatory requirements or TestAmerica's Ethics Policy (Document No. CA-L-P-001). TestAmerica shall not enter any client agreements that conflict with regulatory requirements in the jurisdiction in which the work is performed. Where documented client agreements conflict with this document, but meet the regulatory requirements of the jurisdiction in which the work is performed, the client agreements shall supersede requirements in this CQMP.

TestAmerica Policies, as directed in the CQMP, are documented & adhered to by each analytical testing facility. The Quality Assurance (QA) Manager at each facility is responsible to ensure that their QAM remains in the Corporate-approved format and that all updates are in accordance to the CQMP and their operational processes.

TestAmerica operates under the regulations and guidelines of the following federal programs:

- ❖ Air Force Center for Environmental Excellence (AFCEE)
- ❖ US Army Corp of Engineers, Hazardous, Toxic and Radioactive Waste (USACE HTRW)
- ❖ Clean Air Act (CAA)
- ❖ Clean Water Act (CWA)
- ❖ Comprehensive Environmental Response, Compensation, and Liability Act (CERCLA)
- ❖ Department of Energy (DOE)
- ❖ Marine Protection, Research, and Sanctuaries Act (MPRSA)
- ❖ Navy Facilities Engineering Service Center (NFESC)
- ❖ National Pollutant, Discharge, and Elimination System (NPDES)
- ❖ Nuclear Regulatory Commission (NRC)
- ❖ Occupational Safety and Health Administration (OSHA)
- ❖ Resource Conservation and Recovery Act (RCRA)
- ❖ Safe Drinking Water Act (SDWA)
- ❖ Toxic Substances Control Act (TSCA)

TestAmerica also provides services under various state and local municipal guidelines. A listing of each laboratory's service offerings and certifications is presented on TestAmerica's website or available from the laboratory.

This CQMP was written to comply with the National Environmental Laboratory Accreditation Conference (NELAC) standards.

### **3.5 Terms and Definitions**

A Quality Assurance Program is a company-wide system designed to ensure that data produced by our laboratories conform 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.

## **4.0 Organization and Management**

### **4.1 Roles and Responsibilities**

TestAmerica's organizational structure is presented in Figure 4-1. Corporate employees are located at various TestAmerica facilities as outlined in the organizational structure. A QA Manager shall be designated at each TestAmerica laboratory.

#### **President / CEO**

The President / CEO is a member of the Board of Directors and is ultimately responsible for the quality and performance of all TestAmerica facilities. The President/CEO establishes the overall quality standard and data integrity program for the Analytical Division, providing the necessary leadership and resources to assure that the standard and integrity program are met. The President authorizes the CQMP and as such, sets the standards for the quality system.

#### **Chief Operating Officer (COO)**

The COO serves as the ranking executive for all respective analytical laboratory operational functions and reports to the President/CEO of the Analytical Division. The COO is responsible for the daily management of all analytical laboratories, long-term planning and development of technical policies and management plans. The COO ensures the attainment of corporate objectives through the selection, development, motivation, and evaluation of top management personnel. The COO approves all operating budgets and capital expenditures. The COO authorizes the CQMP and is responsible for ensuring that business and technical operations are conducted in accordance with its requirements.

#### **Vice President of Quality and Technical Services (QTS)**

The Vice President (VP) of Quality and Technical Services is responsible for offerings to clients including risk management, technical assistance, legal compliance and contract administration. The VP of Quality and Technical Services provides support and direction to the Managers of these areas, and supports the COO in decisions regarding long term planning, resource allocation and capital expenditures. The VP QTS authorizes the CQMP and responsibilities include authorization of Manuals, Policies and Procedures, providing support and direction to the Managers of these areas, and supporting the COO in decisions regarding long term planning, resource allocation, and capital expenditures.

### **Director of Quality & Client Advocacy**

The Director of Quality & Client Advocacy reports directly to the VP of Quality & Technical Services. With the aid of the Senior Management Team, Laboratory Directors, Quality Directors, EHS Director, QA Managers and EHS Coordinators, the Director of Quality & Client Advocacy has the responsibility for the establishment, general overview and Corporate maintenance of the Quality Assurance and Environmental, Health and Safety Program within TestAmerica. Additional responsibilities include:

- Review of QA/QC aspects of Corporate SOPs & Policies, national projects and expansions or changes in services.
- Work with various organizations outside of TestAmerica to further the development of quality standards and represent TestAmerica at various trade meetings.
- With the assistance of the EHS Director, development and implementation of the TestAmerica Environmental, Health and Safety Program.

### **Director of Technical Services**

The Director of Technical Services is responsible for establishing, implementing and communicating TestAmerica's Analytical Division's Technical Policies, SOPs, and Manuals. Other responsibilities include conducting technical assessments as required, acting as a technical resource in national contracts review, coordinating new technologies, establishing best practices, advising staff on technology advances, innovations, and applications.

### **General Manager (GM)**

Each GM reports directly to the 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.

### **Quality Directors**

The Quality Directors report to the Director of Quality & Client Advocacy. Together with the Director of Quality & Client Advocacy, the Quality Directors have the responsibility for the establishment, general overview and maintenance of the Analytical Division's Quality Assurance Program within TestAmerica. The Quality Directors are responsible for:

- Oversight of the QA/QC programs within each laboratory. This includes a review of each laboratory-specific QAM and receipt of each laboratory's QA monthly report.
- Review of QA/QC aspects of national projects.
- Assistance with certification & accreditation activities.
- Preparation of a monthly report that includes quality metrics across the Analytical Division and a summary of any quality related initiatives and issues.

### **Ethics and Compliance Officers (ECOs)**

TestAmerica has designated two senior members of the Corporate staff to fulfill the role of Ethics and Compliance Officer (ECO) – VP of Quality & Technical Services and the Director of Quality & Client Advocacy. Each ECO acts as a back-up to the other ECO and both are involved when data investigations occur. Each ECO has a direct line of communication to the entire senior Corporate and lab management staff.

The ECOs ensure that the organization distributes the data integrity and ethical practices policies to all employees and ensures annual trainings and orientation of new hires to the ethics program and its policies. The ECOs are responsible for establishing a mechanism to foster employee reporting of incidents of illegal, unethical, or improper practices in a safe and confidential environment.

The ECOs monitor and audit procedures to determine compliance with policies and to make recommendations for policy enhancements to the CEO, COO, GMs, Laboratory Directors or other appropriate individuals within the laboratory. The ECOs will assist the laboratory QA Manager in the coordination of internal auditing of ethical policy related activities and processes within the laboratory, in conjunction with the laboratories regular internal auditing function.

The ECOs will also participate in investigations of alleged violations of policies and work with the appropriate internal departments to investigate misconduct, remedy the situation, and prevent recurrence of any such activity.

### **Chief Information Officer (CIO)**

The CIO is responsible for establishing, implementing and communicating TestAmerica's Information Technology (IT) Policies, SOPs and Manuals. Other responsibilities include coordinating new technologies, development of electronic communication tools such as TestAmerica's intranet and internet sites, ensuring data security and documentation of software, ensuring compliance with the NELAC standard, and assistance in establishing, updating, and maintaining Laboratory Information Management Systems (LIMS) at the various TestAmerica facilities.

### **Environmental Health and Safety Director (EHS)**

The EHS Director reports directly to the Director of Quality & Client Advocacy. The EHS Director is responsible for the development and implementation of the TestAmerica Environmental, Health and Safety program. Responsibilities include:

- Consolidation and tracking all safety and health-related information and reports for the company, and managing compliance activities for TestAmerica locations.
- Coordination/preparation of the Corporate Environmental Health and Safety Manual that is used by each laboratory to prepare its own laboratory-specific Safety Manual.
- Preparation of information and training materials for laboratory EHS Coordinators.
- Assistance in the internal and external coordination of employee exposure and medical monitoring programs to insure compliance with applicable safety and health regulations.
- Serving as Department of Transportation (D.O.T.) focal point and providing technical assistance to location management.
- Serving as Hazardous Waste Management main contact and providing technical assistance to location management.

### **Laboratory Director**

The Laboratory Director oversees the daily operations of the laboratory. The Laboratory Director's responsibilities include supervision of staff, setting goals and objectives for both the business and the employees, and achieving the financial, business, technical and quality objectives of the facility. The Laboratory Director ensures timely compliance with audits and corrective actions, and is responsible for maintaining a working environment which encourages open, constructive problem solving and continuous improvement.

### **QA Manager**

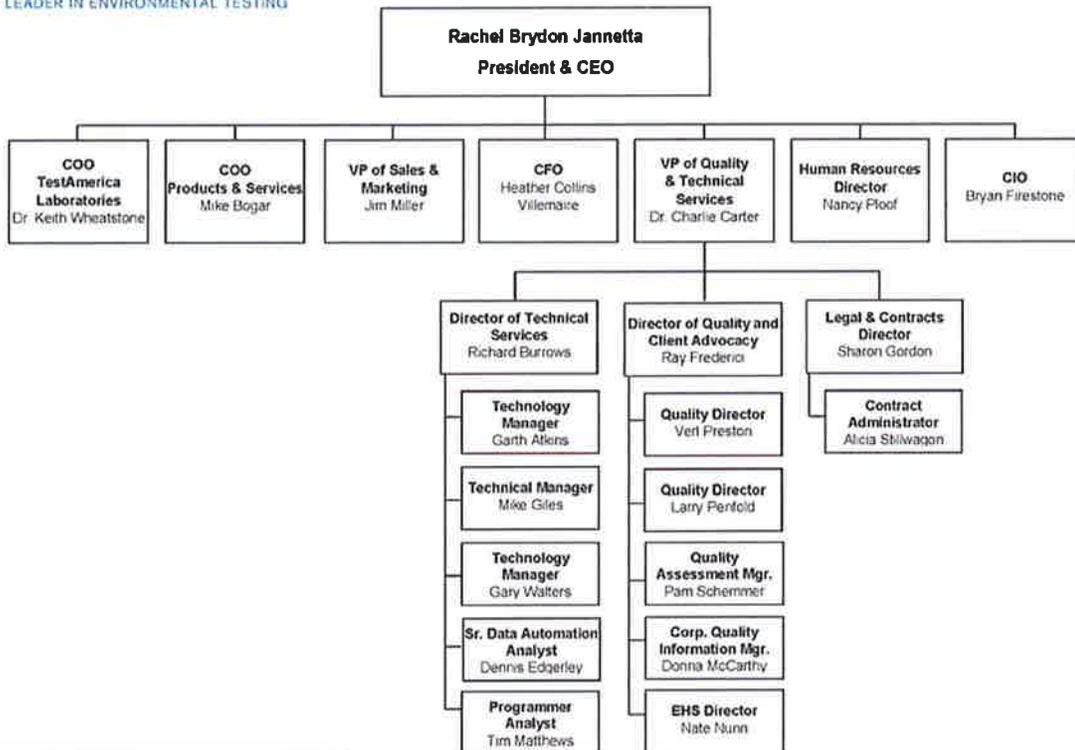
The QA Manager is responsible for ensuring that the laboratory's quality system and QAM meet the requirements set forth in the CQMP, providing quality systems training to all new personnel, maintaining a QAM, and performing or overseeing systems, data, special, and external audits. The QA Manager performs, or supervises, the maintenance of QA records, the maintenance of certifications and accreditations, the submission of monthly QA Reports, and assists in reviewing new work as needed. The QA Manager shall have the final authority to accept or reject data, and to stop work in progress in the event that procedures or practices compromise the validity and integrity of analytical data. The QA Manager is available to any employee at the facility to resolve data quality or ethical issues. The QA Manager shall be independent of laboratory operations. The facility QA Manager has an indirect reporting relationship to the Quality Director. Each laboratory's QAM has further descriptions of roles and responsibilities at the facility level.

### **Technical Director**

The Technical Director(s) of a laboratory has overall responsibility for a defined portion of the technical operations of the laboratory, and may or may not be the Laboratory Director. 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 QA, and directs evaluation of new methods.

Figure 4-1.

TestAmerica Organizational Chart



Note: QA Managers and Safety Coordinators in all laboratories and facilities have a dotted line reporting relationship to Corporate QA and EHS.

Jan 2009

## **5.0 Quality System**

### **5.1 Quality Assurance Policy**

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.

### **5.2 Management Commitment to Quality Assurance and Data Integrity**

TestAmerica management is committed to providing data of known and documented quality and the best service in the environmental testing industry. To ensure that the data produced and reported by TestAmerica meet the requirements of its clients and comply with the letter and spirit of municipal, state and federal regulations, TestAmerica maintains quality and data integrity systems that are clear, effective, well communicated, and supported at all levels in the company.

### **5.3 Objectives of the Quality System**

The goal of the TestAmerica quality system is to ensure that business and technical operations are conducted with the highest standards of professionalism and ethics in the industry.

To achieve this goal, it is necessary to provide TestAmerica clients with not only scientifically sound, well-documented, and regulatory-compliant data, but also to ensure that TestAmerica provides the highest quality service available in the industry with uncompromising data integrity. A well-structured and well-communicated quality system is essential in meeting this goal. TestAmerica's quality system is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

This CQMP is the basis for TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica analytical facilities shall conduct their operations.

**5.3.1 Laboratory Quality Assurance Manual (QAM)**

Each TestAmerica analytical facility shall have a Quality Assurance Manual that further describes the specific QA program at the laboratory.

The Quality Assurance Manual shall address the following:

Section No.	Title
-	Cover Page
1.0	Title Page
2.0	Table of Contents
3.0	Introduction
4.0	Organization & Management (NELAC 5.4.1)
5.0	Quality Systems (NELAC 5.4.2)
6.0	Document Control (NELAC 5.4.3)
7.0	Service to the Client (NELAC 5.4.7)
8.0	Subcontracting of Tests (NELAC 5.4.5)
9.0	Purchasing Services & Supplies (NELAC 5.4.6)
10.0	Complaints (NELAC 5.4.8)
11.0	Control of Non-Conforming Work (NELAC 5.4.9)
12.0	Corrective Action (NELAC 5.4.10)
13.0	Preventive Action (NELAC 5.4.11)
14.0	Control of Records (NELAC 5.4.12)
15.0	Audits (NELAC 5.4.13)
16.0	Management Reviews (NELAC 5.4.14)
17.0	Personnel (NELAC 5.5.2)
18.0	Accommodations & Environmental Conditions (NELAC 5.5.3)
19.0	Test Methods & Method Validation (NELAC 5.5.4)
20.0	Equipment (and Calibration) (NELAC 5.5.5)
21.0	Measurement Traceability (NELAC 5.5.6)
22.0	Sampling (NELAC 5.5.7)
23.0	Handling of Samples (NELAC 5.5.8)
24.0	Assuring the Quality of Test Results (NELAC 5.5.9)
25.0	Reporting Results (NELAC 5.5.10)

**5.3.2 Data Quality Objectives**

The Data Quality Objectives (DQO) process is a methodology used by project planners to define the environmental question to be answered and the processes needed to ensure the generation of the type, quantity, and quality of environmental data that will be needed for the intended application. The process results in a series of qualitative and quantitative statements termed Data Quality Objectives (DQOs). DQOs are identified before project initiation, and are the basis of laboratory quality control limits in project documents, such as Quality Assurance Program Plans (QAPPs) and Sampling & Analysis Plans (SAPs). QC samples routinely used by TestAmerica are described in Section 24.

Data quality indicators often defined as DQOs include precision, accuracy, representativeness, completeness, and comparability:

**Precision** is the degree to which a set of observations or measurements of the same property, obtained under similar conditions, agree with each other. At the client project level, precision is

usually expressed as standard deviation, variance or range, in either absolute or relative terms. In laboratory reports, batch precision is commonly expressed in terms of relative percent difference (RPD) for replicate pairs of measurements (e.g., matrix spike / matrix spike duplicates) or relative standard deviation (RSD) for more than two replicates.

**Accuracy** is 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 that are due to sampling and analytical operations; a data quality indicator. Accuracy is commonly expressed by the laboratory as the percent recovery of analytical spikes. However, project teams typically consider bias and precision separately when assessing data quality.

**Representativeness** is the degree to which data characterizes a population being studied, such as a sampling point or an environmental decision unit. Data representativeness is primarily a function of sampling strategy (e.g., the sampling scheme must be designed to maximize representativeness). The portion of the sample used for analysis must also be representative of the entire sample delivered to the laboratory. However, due to the lack of industry-wide agreement on a more quantifiable definition, this term is going out of favor.

**Completeness** is defined as the percentage of measurements that are judged valid or useable. Factors negatively affecting completeness include the following: sample leakage or breakage in transit or during handling, loss of sample during laboratory analysis through accident or improper handling, improper documentation such that traceability is compromised, or sample result is rejected due to failure to conform to QC specifications. A completeness objective of greater than 90% of the data specified by the statement of work is the goal established for most projects.

**Comparability** is a measure of the confidence with which one data set can be compared to another. To ensure comparability, project plans typically require the use of methods approved by EPA or other standards setting bodies. Within the laboratory, analysts are required to use uniform procedures (e.g., SOPs) and a uniform set of units and calculations for analyzing and reporting environmental data.

What is most important for the laboratory is that the components of analytical variability (uncertainty) can be estimated when QC samples of the right types and at the appropriate frequency are incorporated into measurement process at the analytical laboratory. With these QC results, the laboratory's client can assess whether or not the DQOs were met. With data of known and documented quality, the laboratory data and ultimately the environmental decision made using the data can withstand scientific and legal scrutiny.

## **6.0 Document Control**

### **6.1 Document Type**

The following documents, at a minimum, must be controlled at each TestAmerica laboratory:

- ❖ Laboratory Quality Assurance Manual (QAM)
- ❖ Standard Operating Procedures (SOPs)
- ❖ Corporate Quality Management Plan (CQMP)
- ❖ Corporate Policies and Procedures

❖ Corporate Quality Policy Memorandums

**6.2 Document Control Procedure**

Security and control of documents are necessary to ensure that confidential information is not distributed and that all current copies of a given document are from the latest applicable revision. Unambiguous identification of a controlled document is maintained by identification of the following items in the document header: Document Number, Revision Number, Revision Date, Effective Date, and Number of Pages. Controlled documents are authorized by Management and/or the QA Department. Controlled documents are marked as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

Controlled documents shall be available at all locations where the operational activity described in the document is performed.

**6.3 Document Revision**

Quality system policies and procedures will be reviewed at a minimum of every two years<sup>1</sup> and revised as appropriate. Changes to documents occur when a procedural change warrants a revision of the document. When an approved revision of a controlled document is ready for distribution, obsolete copies of the document shall be replaced with the current version of the document. The previous revision of the controlled document must be archived by the QA Department.

<sup>1</sup> Laboratory's participating in the Department of Defense (DoD) programs will update their documents every calendar year. Corporate quality documents that support the DoD programs will be reviewed annually by Corporate staff members.

**6.4 Official Documents**

The TestAmerica Corporate Operations staff posts Corporate Manuals, Standard Operating Procedures, Policies, Quality Policy Memorandums, Work Instructions, White Papers and Training Materials on TestAmerica's intranet site. These are collectively termed "Official Documents" and encompass the Policies and Procedures that all testing facilities are required to employ. Corporate Quality Policy Memorandums are employed to notify personnel of required changes and subsequent implementation to both administrative and technical issues. By reference, these memorandums can be attached to Official Documents but do not need to be included in these documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate Documents is found in the Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archive.

**7.0 Service to the Client**

**7.1 Contract Review**

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 TestAmerica's intent to provide both standard and customized environmental testing services to

our clients. To ensure project success, technical staff shall perform a thorough review of technical and QC requirements contained in contracts. Contracts are reviewed for adequately defined requirements and TestAmerica's capability to meet those requirements.

Contract review shall include a review of the client's requirements in terms of compound lists, test methodology requested, sensitivity, accuracy, and precision requirements. The TestAmerica representative ensures that the laboratory's test methods are suitable to achieve these requirements and must ensure that the laboratory holds the appropriate certifications and approvals to perform the work. The review also includes the laboratory's capabilities in terms of turnaround time, capacity, and resources to provide the services requested, as well the laboratory's ability to provide the documentation, whether hardcopy or electronic. If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica laboratory or to an outside firm, this must be documented and discussed with the client prior to contract approval (refer to Section 8, Subcontracting).

All contracts entered into by TestAmerica shall be reviewed and approved by the appropriate personnel at the facility or facilities performing the work. Any contract requirement or amendment to a contract communicated to TestAmerica verbally must be documented and confirmed with the client in writing. Any discrepancy between the client's requirements and TestAmerica's capability to meet those requirements is resolved in writing before acceptance of the contract. Contract amendments, initiated by the client and/or TestAmerica, are documented in writing for the benefit of both the client and TestAmerica.

All contracts, Quality Assurance Program Plans (QAPPs), Sampling & Analysis Plans (SAPs), contract amendments, and documented communications become part of the permanent project record as defined in Section 14.

## **7.2 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, TestAmerica assigns a Project Manager (PM) to each client. The PM is the primary point of contact for the client. It is the PM's responsibility to ensure that project-specific technical and QC requirements are effectively communicated to the laboratory personnel before and during the project.

Each TestAmerica laboratory shall have established project planning procedures in order to ensure that communication is inclusive and effective. These include project memos, designation and meetings of project teams, and meetings between the laboratory staff and the client. TestAmerica has found it very effective to invite the client into this process. TestAmerica strongly encourages our clients to visit the laboratories and hold formal or informal sessions with employees in order to effectively communicate ongoing client needs as well as project-specific details for customized testing programs.

## **7.3 Client Confidentiality**

Data and sample materials provided by the client or at the client's request, and the results obtained by TestAmerica, shall be held in confidence (unless such information is generally available to the public or is in the public domain or client has failed to pay TestAmerica for all services rendered or is otherwise in breach of the terms and conditions set forth in the TestAmerica and client contract) subject to any disclosure required by law or legal process.

TestAmerica's reports, and the data and information provided therein, are for the exclusive use and benefit of the client, and are not released to a third party without written consent from the client

#### **7.4 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 develop lab and client specific surveys to assess client satisfaction.

### **8.0 Subcontracting**

Subcontracting must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. 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. The originating laboratory shall obtain proof of certification from the subcontract facility, and retain this information in TestAmerica records. Where applicable, specific QC guidelines, QAPPs, and/or SAPs are transmitted to the subcontract laboratory. Samples are subcontracted under formal Chain of Custody (COC). It is not acceptable to subcontract work outside of TestAmerica without attempting to negotiate alternative requirements with the client and/or the proposed TestAmerica subcontract lab.

Non-TestAmerica subcontract laboratories may receive an on-site audit by a representative of TestAmerica's QA staff if it is deemed appropriate by the QA Manager. The audit involves a measure of compliance with the required test method, QC requirements, as well as any special client requirements. The originating laboratory may also perform a paper audit of the subcontractor, which could entail reviewing the QAM, the last two PT studies, and a copy of any recent regulatory audits with the laboratory's responses. Complete details on TestAmerica's Subcontracting Procedure are available in Corporate SOP No. CA-L-S-002.

Intra-company subcontracting within TestAmerica must be arranged with the documented consent of the client, in a timely response which shall not be unreasonably refused. The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs.

Project reports from both TestAmerica and external subcontractors are discussed in Section 25.

### **9.0 Purchasing Services and Supplies**

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.

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Purchasing guidelines for equipment and reagents meet with the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with the Corporate SOP No. CA-Q-S-001, Solvent & Acid Lot Testing & Approval.

## **10.0 Complaints**

TestAmerica believes that an effective client complaint handling process has important business and strategic value. Listening to and documenting client's concerns captures "client knowledge" that helps to continually improve the process and outpace the competition. Implementing a client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

Client complaints shall be documented, communicated to management, and addressed promptly and thoroughly. Client complaints are documented by the employee receiving the complaint. The documentation can take the form of a corrective action report (as described in Section 12) or in a format specifically designed for that purpose.

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 is notified and may conduct a special audit to assist in resolving the issue. A written confirmation, or letter to the client, outlining the issue and response taken is strongly recommended as part of the overall action taken.

The number of client complaints shall be reported to the Quality Directors in the QA monthly report submitted by each laboratory. The overall number of complaints received per facility is tracked and the appropriateness of the response to client complaints is assessed. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Management Systems Review. Most client feedback comes either verbally or in writing to TestAmerica employees. However, TestAmerica also uses a number of additional mechanisms to obtain client feedback including a customer satisfaction survey and a response card system. Each of these is monitored for trends and opportunities for improvement.

## **11.0 Control of Non-Conforming Work**

Each laboratory shall have a procedure to control and document non-conformances. Non-conformances broadly include any QC result outside of established control limits or actions outside of established processes. Non-conformances may relate to client specific requirements, procedural requirements, or equipment issues. All non-conformances in the laboratory are documented at the time of their occurrence.

All non-conformances that affect a sample and/or sample data become part of the affected project's permanent record. When appropriate, reanalysis is performed where QC data falls outside of specifications, or where data appears anomalous. If the reanalysis is within established tolerances, the results are approved. If the reanalysis is still outside tolerances, further reanalysis or consultation with the Supervisor, Manager, PM, Laboratory Director, or QA Manager for direction may be required. All records of reanalysis are kept with the data files.

Where non-conformances specifically affect a client's sample and/or data, the client shall be informed and action must be taken. Action can take the form of reporting and flagging the data, and including a description of the non-conformance in the project narrative or cover letter.

## **12.0 Corrective Action**

### **12.1 General**

Each TestAmerica laboratory shall maintain an established, documented corrective action process. Each corrective action is thoroughly investigated. The outcome of the investigation, actions taken, and follow-up is documented. The more significant the issue to be corrected, the more formal the investigation into root cause and the more detailed the documentation that is required.

### **12.2 Initiation**

Any employee in TestAmerica shall be authorized to initiate a corrective action. The initial source of corrective action can also be external to TestAmerica (e.g., corrective action due to client complaint, regulatory audit, or proficiency test). When a problem that requires corrective action is identified, the following items are identified by the initiator on the corrective action report (or however named): the nature of the problem, the name of the initiator, and the date. If the problem relates to a specific client project, the name of the client and laboratory project number is recorded, and the PM is informed immediately.

### **12.3 Cause Analysis**

The corrective action process must be embarked upon as a joint, problem solving, and constructive effort. Identification of systematic errors, or errors that are likely to occur repetitively due to a defect or weakness in a system, is particularly valuable in maintaining an environment of continuous improvement in laboratory operations.

When a corrective action report (or however named) is initiated, the initiator works with the affected employee(s) and/or department(s) to identify the root cause of the problem. An essential part of the corrective action process is to identify whether the problem occurred due to a systematic or isolated error.

If the initiator of the corrective action report (or however named) is uncertain as to what would constitute appropriate corrective action or is unable to resolve the situation, the problem is identified to the Supervisor, Manager, Laboratory Director or the QA Manager who provides assistance in the corrective action process. The root cause of the problem and associated cause analysis is documented.

### **12.4 Corrective Action**

Once the root cause of a problem is identified, the initiator and affected employee(s) and/or department(s) examine potential actions that will rectify the present problem to the extent possible, and prevent recurrence of future, similar occurrences. An appropriate corrective action is then recommended. The corrective action must be appropriate for the size and nature of the issue.

If the corrective action concerns a specific project related issue, the PM or Customer Service Manager approves the corrective action before its implementation.

Implementation of the corrective action and the date of implementation are documented on the corrective action report (or however named).

If a corrective action is related to a specific project report, it is included in the project file. An essential part of the corrective action process is communication and awareness of the problem, the cause, and the action taken to prevent future occurrences and/or rectify the immediate problem.

### **12.5 Monitoring Corrective Action**

The QA department reviews corrective action reports and selects one or more of the more significant corrective actions for inclusion in the annual systems audit. The QA Department also may implement a special audit. The purpose of inclusion of the corrective action process in both routine and special audits is to monitor the implementation of the corrective action and to determine whether the action taken has been effective in overcoming the issue identified.

## **13.0 Preventative Action**

Each laboratory shall maintain an established, documented preventative action process. Preventative action is identifying process weaknesses which have the potential to lead to failure(s). Preventative action includes analysis of the quality system to detect, analyze, and eliminate potential causes of non-conformances. It may include trend analysis using control charts to detect chemical analysis problems before QC results exceed control limits at a high frequency. When potential problems are identified, preventative action is initiated to effectively address the problem to eliminate or reduce the risk identified.

### **13.1 Management of Change**

A Management of Change System is a documentation system designed to manage significant events and changes that occur within the laboratory. The types of changes include, but are not limited to: facility changes, major accreditation & approval changes, addition or deletion to laboratory capabilities, key personnel changes and the addition of a new type of instrumentation. Through a documentation system (however named by the laboratories), 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.

A Management of Change System can apply to all areas except in the application of: maintenance, repairs and activities which are "repair or replacement in-kind", and other changes at the discretion of the Laboratory Director. A laboratory may expand on this process for internal changes as long as the basic framework of documentation & communication is followed.

## 14.0 Control of Records

### 14.1 Record Types & Record Retention

Table 14-1 outlines TestAmerica's standard record retention time. 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 14.2.

**Table 14-1. Example of TestAmerica Record Types<sup>1</sup>**

	<u>Record Types<sup>1</sup>:</u>	<u>Retention Time:</u>
<b>Technical Records</b>	<ul style="list-style-type: none"> <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*
<b>Official Documents</b>	<ul style="list-style-type: none"> <li>- Quality Assurance Manual (QAM)</li> <li>- Work Instructions</li> <li>- Policies</li> <li>- SOPs</li> <li>- Policy Memorandums</li> <li>- Manuals</li> </ul>	5 Years from document retirement date*
<b>QA Records</b>	<ul style="list-style-type: none"> <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*  <b>Data Investigation:</b> 5 years or the life of the affected raw data storage whichever is greater (beyond 5 years if ongoing project or pending investigation)
<b>Project Records</b>	<ul style="list-style-type: none"> <li>- Sample Receipt &amp; COC 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*
<b>Administrative Records</b>	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	7 years
	Technical Training Records	

- <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 are listed in each facility QAM.

**Table 14-2. Example: Special Record Retention Requirements**

<b>Program</b>	<b>Retention Requirement <sup>1</sup></b>
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)	10 years
NY Potable Water NYCRR Part 55-2	10 years
Ohio VAP	10 years and State contacted prior to disposal
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup> Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

#### **14.2 Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the TestAmerica standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement must be implemented and noted in the archive or addressed in a facility-specific records retention procedure. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the record retention management system provides information as to who to contact for authorization prior to destroying the data.

#### **14.3 Archives and Record Transfer**

Archives must be indexed such that records are accessible on either a project or temporal basis. Archives are protected against fire, theft, loss, deterioration, and vermin. Electronic records are protected from deterioration caused by magnetic fields and/or electronic deterioration. Access to archives is controlled and documented. On-site and/or off-site facilities may be used.

TestAmerica ensures that all records are maintained as required by the regulatory guidelines and per the CQMP upon facility location change or ownership transfer. Upon a laboratory location change, all archives are retained by TestAmerica in accordance with the CQMP. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established.

## 15.0 Audits

### 15.1 Internal Audits - Audit Types and Frequency

A number of types of audits shall be performed at TestAmerica. Audit type and frequency are categorized in Table 15-1.

**Table 15-1. Types of Internal Audits and Frequency**

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	- All SOPs within a 2-year period - All new analysts or new analyst/methods within 3 months of IDOC
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

#### 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, 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 SOP Method Compliance**

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 Performance Testing (PT)**

The laboratory participates semi-annually (NELAC) or annually (Non-NELAC) 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**

TestAmerica facilities are routinely audited by clients and external regulatory authorities. TestAmerica is available for these audits and makes every effort to provide the auditors with the personnel, documentation, and assistance required by the auditors. TestAmerica recommends that the audits be scheduled with the QA Department so that all necessary personnel are available on the day of the audit.

### **15.3 Audit Findings**

Audit findings are documented using the corrective action process. 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 be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the laboratory management personnel 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.

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.

## **16.0 Management Reviews**

### **16.1 QA Reports to Management**

A monthly QA report shall be prepared by the QA Manager or their designee and forwarded to their Laboratory Director, GM and Quality Director. The report includes statistical results that are used to assess the effectiveness of the quality system.

A Corporate QA Monthly Report containing a compilation of the Facility QA reports statistics, information on progress of the Corporate QA program, and a narrative outlining significant occurrences and/or concerns shall be compiled by the Quality Directors and forwarded to the Director of Quality & Client Advocacy who, after preparing comments, forwards the report to the COO, VP of QTS, GMs and the entire Senior Management Team.

#### **16.1.1 Monthly QA Report and Metrics**

The QA Manager's monthly QA report is due by the fifth day of the month. The report will contain a narrative summary and metrics spreadsheet. At a minimum, the report content will contain the laboratory's status for defined quality metrics and a discussion of both improvements and weaknesses in the quality system. During the course of the year, the Laboratory Director, General Manager, Director of Quality & Client Advocacy or the Quality Director may request that additional information be added to the report.

## **16.2      Management Systems Review**

Each laboratory shall perform a management quality system review annually in accordance with the Corporate SOP No. CA-Q-S-008, Quality Systems Management Review. This will synchronize quality planning with fiscal year planning. The management quality system review will assess the adequacy of the laboratory's quality system and plan any changes in laboratory organization, policies, practices, certifications, accreditations in order to achieve operational efficiencies, meet regulatory requirements and client expectations.

## **17.0      Personnel**

### **17.1      General**

TestAmerica management believes that its highly qualified, ethical and professional staff is the single most important aspect in assuring the highest level of data quality and service in the industry.

TestAmerica staff consists of over two thousand professionals and support personnel that include, but not limited to, the following positions:

- ❖ General Manager
- ❖ Customer Service Manager
- ❖ Quality Assurance (QA) Manager
- ❖ Laboratory Director
- ❖ Technical Director
- ❖ Department Supervisor
- ❖ Information Technology Manager
- ❖ Human Resources Manager
- ❖ Project Manager
- ❖ Department Manager
- ❖ Analyst
- ❖ Sample Custodian
- ❖ Technician
- ❖ Quality Assurance Specialist
- ❖ Data Review Specialist
- ❖ Information Technology Specialist

#### **17.1.1    Training**

TestAmerica is committed to furthering the professional and technical development of employees at all levels. Minimum training requirements for TestAmerica employees are outlined in Table 17-1.

**Table 17-1. TestAmerica Employee Minimum Training Requirements**

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

*\*From date of initial employment unless otherwise indicated.*

Technical training is accomplished within each laboratory by management to ensure method comprehension. All new personnel shall be required to demonstrate competency in performing a particular method by successfully completing an Initial Demonstration of Capability (DOC) before conducting analysis independently on client samples.

DOCs are performed by analysis of four replicate QC samples. Results of successive LCS analyses can be used to fulfill the DOC requirement. The accuracy and precision, measured as average recovery and standard deviation (using n-1 as the population), of the 4 replicate results are calculated and compared to those in the test method (where available). If the test method does not include accuracy and precision requirements, the results are compared to target criteria set by the laboratory. The laboratory sets the target criteria such that they reflect the DQOs of the specific test method or project. A DOC Certification Statement is recorded and maintained in the employee's training or personnel file. Figure 17-1 shows an example of a DOC Certification Statement.

The following evidence must be on file at the laboratory for each technical employee. Additional items may be kept on file based on the laboratory-specific QAM.

- ❖ DOC.
- ❖ The employee has read and understood the latest version of the laboratory's quality documentation.
- ❖ The employee has read and understood the latest, approved version of all test methods and/or SOPs for which the employee is responsible.
- ❖ Annual evidence of continued DOC that may include successful analysis of a blind QC sample on the specific test method, or a similar test method, or an annual DOC, or four successive & successful LCSs.
- ❖ An Ethics Agreement signed by each staff member (renewed each year).

**Figure 17-1. Example Demonstration of Capability Certification Statement**

Laboratory Name: _____							
Laboratory Address: _____							
Method: _____				Matrix: _____			
Date: _____		Analyst(s): _____					
Source of Analyte(s): _____							
			Analytical Results				
Analyst	Conc. (Units)	Rep 1	Rep 2	Rep 3	Rep 4	Avg. % Recovery	% RSD
_____	_____	_____	_____	_____	_____	_____	_____
<p>% RSD = Percent relative standard deviation = standard deviation divided by average % Recovery</p> <p>Raw data reference: _____</p> <p><b>Certification Statement:</b></p> <p>We, the undersigned, certify that:</p> <ol style="list-style-type: none"> <li>1. The cited test method has met Demonstration of Capability requirements.</li> <li>2. The test method was performed by the analyst(s) identified on this certification.</li> <li>3. A copy of the test method and the laboratory-specific SOPs are available for all personnel on site.</li> <li>4. The data associated with the method demonstration of capability are true, accurate, complete, and self-explanatory.</li> <li>5. All raw data necessary to reconstruct and validate these analyses have been retained at the facility, and the associated information is well organized and available for review.</li> </ol>							
_____ Analyst Signature				_____ Date			
_____ Technical Director Signature				_____ Date			
_____ Quality Assurance Manager Signature				_____ Date			

**17.1.2 Ethics Policy**

Establishing and maintaining a high ethical standard is an important element of a quality system. In order to ensure that all personnel understand the importance the company places on maintaining high ethical standards at all times, TestAmerica has established an *Ethics Policy*, Policy No. CA-L-P-001, and an Ethics Agreement. Each employee shall sign the Ethics Agreement, signifying agreed compliance with its stated purpose. The ethics agreement is required to be re-signed on an annual basis.

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 the Company's ability to do work on Government contracts, and for that reason, the Company has a Zero Tolerance approach to such violations.

Ethics is also a major component of TestAmerica's quality and data integrity systems. Each employee must be introduced to TestAmerica's Ethics Policy within 1 week of hire; and receive the Comprehensive Ethics training and Quality Training within 90 days of hire. Technical employees shall also receive Data Integrity Training within 30 days of hire. Annually, Ethics Refresher Training will be provided. Employees must be trained as to the legal and

environmental repercussions that result from data misrepresentation. A data integrity hotline is maintained by TestAmerica and administered by the Corporate Quality Department.

## **18.0 Accommodations & Environmental Conditions**

Each laboratory must be secure and access must be controlled and documented. Access is controlled by various measures including locked doors, passwords, electronic access cards, security codes, and staffed reception areas. All visitors sign in and are escorted by TestAmerica personnel while at a laboratory.

TestAmerica's facilities are designed for efficient, automated high-quality operations. All laboratories are equipped with Heating, Ventilation, and Air Conditioning (HVAC) systems appropriate to the needs of environmental testing laboratories. Environmental conditions in the facilities, such as hood flow, are routinely monitored and documented.

All TestAmerica facilities are 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. TestAmerica also provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, respirators, etc..

## **19.0 Test Methods and Method Validation**

### **19.1 Test Methods**

Most of the test methods performed at TestAmerica originate from test methods published by a regulatory agency such as the U.S. EPA and other state and federal regulatory agencies. These include, but are not limited to, the following published compendiums of test methods:

- ❖ Prescribed Procedures for Measurement of Radioactivity in Drinking Water, EPA-600/4-80-032, August 1980.
- ❖ Eastern Environmental Radiation Facility Radiochemistry Procedures Manual, EPA, PB84-215581, June 1984.
- ❖ HASL-300 28th Edition, Environmental Measurements Laboratory (EML), 1997.
- ❖ Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fourth Edition, EPA/600/4-90/027F, August 1993.
- ❖ Methods for Measuring the Acute Toxicity of Effluents and Receiving Waters to Freshwater and Marine Organisms, Fifth Edition, EPA-821-R-02-012, October 2002.
- ❖ Short-Term Methods for Estimating the Chronic Toxicity of Effluents and Receiving Waters to Freshwater Organisms, Fourth Edition, EPA-821-R-02-013, October 2002.
- ❖ Analytical Method for Determination of Asbestos Fibers in Water, EPA-600/4-83, September 1983.
- ❖ Determination of Asbestos Structures Over 10-mm in Length in Drinking Water, EPA-600/R-94-134, June 1994.
- ❖ Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, US EPA, January 1996.

- ❖ Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act, and Appendix A-C; 40 CFR Part 136, USEPA Office of Water, Revised as of March 12, 2007, Appendix A to Part 136 - Methods for Organic Chemical Analysis of Municipal and Industrial Wastewater (EPA 600 Series)
- ❖ Methods for Chemical Analysis of Water and Wastes, EPA 600 (4-79-020), 1983.
- ❖ Methods for the Determination of Inorganic Substances in Environmental Samples, EPA-600/R-93/100, August 1993.
- ❖ Methods for the Determination of Metals in Environmental Samples, EPA/600/4-91/010, June 1991. Supplement I: EPA-600/R-94/111, May 1994.
- ❖ Methods for the Determination of Organic Compounds in Drinking Water, 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. Supplement III EPA/600/R-95/131 - August 1995 (EPA 500 Series) (EPA 500 Series methods)
- ❖ Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 1994
- ❖ NIOSH Manual of Analytical Methods, 4<sup>th</sup> ed., August 1994.
- ❖ Statement of Work for Inorganics Analysis, ILM04.1, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- ❖ Statement of Work for Inorganics Analysis, ILM05.2, 5.3 & 5.4 USEPA Contract Laboratory Program Multi-media, Multi-concentration
- ❖ Statement of Work for Organics Analysis, OLM04.1, 4.2 & 4.3 USEPA Contract Laboratory Program, Multi-media, Multi-concentration.
- ❖ Statement of Work for Organics Analysis, SOM01.1 & 1.2, USEPA Contract Laboratory Program, Multi-media, Multi-concentration
- ❖ Standard Methods for the Examination of Water and Wastewater, 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.
- ❖ 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; Final Update IV, January 2008.
- ❖ Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- ❖ National Status and Trends Program, National Oceanographic and Atmospheric Administration, Volume I-IV, 1985-1994.
- ❖ Manual for the Certification of Laboratories Analyzing Drinking Water (EPA 815-R-05-004, January 2005)
- ❖ Code of Federal Regulations (CFR) 40, Parts 136, 141, 172, 173, 178, 179 and 261

**19.2 Standard Operating Procedures**

Each laboratory shall maintain a Standard Operating Procedure (SOP) Index for both Method and Process SOPs. Method SOPs are maintained to describe a specific test method. Process SOPs are maintained to describe functions and processes not related to a specific test method.

Technical SOPs may contain the following information (in any particular order):

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 19-1).

Identification of Test Method	Calibration and Standardization
Applicable Matrix	Procedure
Reporting Limit	Calculations
Scope and Application, including test analytes	Method Performance
Summary of the Test Method	Pollution Prevention
Definitions	Data Assessment and Acceptance Criteria for Quality Control Measures
Interferences	Corrective Actions for Out-of-Control Data
Safety	Contingencies for Handling Out-of-Control or Unacceptable Data
Equipment and Supplies	Waste Management
Reagents and Standards	References
Sample Collection, Preservation, Shipment and Storage	Tables, Diagrams, Flowcharts and Validation Data
Quality Control	Method Modifications
	SOP Revision History

Non-Technical SOPs may contain the following information (in any particular order):

Title Page with Document Name, Document Number, Revision Number, Effective Date, Page Numbers and Total # of Pages, Authorized Signatures, Dates and Proprietary Information Statement (Figure 19-1).

Scope	Safety
Summary	Procedure
Definitions	References
Responsibilities	Tables, Diagrams and Flowcharts
	SOP Revision History

The QA Department is responsible for maintenance of SOPs, archival of SOP historical revisions, maintenance of an SOP index, and records of controlled distribution. SOPs, at a minimum, must undergo periodic review as described the each facility's QAM or SOP. Where an SOP is based on a published method, the laboratory must maintain a copy of the reference method.

### Figure 19-1. Proprietary Information Statement

This documentation has been prepared by TestAmerica Laboratories, Inc. and its affiliates ("TestAmerica"), solely for their own use and the use of their customers in evaluating their qualifications and capabilities in connection with a particular project. The user of this document agrees by its acceptance to return it to TestAmerica upon request and not to reproduce, copy, lend, or otherwise disclose its contents, directly or indirectly, and not to use it for any other purpose other than that for which it was specifically provided. The user also agrees that where consultants or other outside parties are involved in the evaluation process, access to these documents shall not be given to said parties unless those parties also specifically agree to these conditions.

**THIS DOCUMENT CONTAINS VALUABLE CONFIDENTIAL AND PROPRIETARY INFORMATION. DISCLOSURE, USE OR REPRODUCTION OF THESE MATERIALS WITHOUT THE WRITTEN AUTHORIZATION OF TESTAMERICA IS STRICTLY PROHIBITED. THIS UNPUBLISHED WORK BY TESTAMERICA IS PROTECTED BY STATE AND FEDERAL LAW OF THE UNITED STATES. IF PUBLICATION OF THIS WORK SHOULD OCCUR THE FOLLOWING NOTICE SHALL APPLY:**

**©COPYRIGHT 2009 TESTAMERICA LABORATORIES, INC. ALL RIGHTS RESERVED.**

#### SOP Appendix

In some cases, a standard laboratory procedure is modified slightly for a specific client or project at the client or regulatory agency's request. In these cases, an Appendix to the SOP may be attached that indicates the modifications to the SOP which are specific to that project. SOP appendices shall not be used to alter test methods required by regulation such that the modifications would result in non-compliance with the regulation. All client- or project-specific modifications must be approved by laboratory management.

### **19.3 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.

#### **Determination of Method Selectivity and Interferences**

If the new method is based on a published consensus method or an EPA method, then analysis of blanks and spikes as described in the source method is sufficient. If the laboratory is developing a method without a published source method, then more extensive validation is required. The laboratory must perform analysis of spikes in each sample matrix of interest. In some cases to achieve the required selectivity for an analyte, a confirmation analysis may be required

#### **Determination of Method Sensitivity**

The sensitivity of new methods is normally demonstrated using the procedure described in the Corporate SOP No. CA-Q-S-006, Detection Limits. Sensitivity can also be estimated for short-term projects using other techniques (e.g., signal-to-noise ratio of low concentration standards), but only with client agreement.

#### **Limit of Quantitation (LOQ) and Reporting Limit (RL)**

The LOQ is the minimum level at which the concentration of an analyte can be determined within limits of confidence required by the data user. The lowest calibration standard must be at

or below the LOQ. The LOQ cannot be at or below the detection limit concentration. Confirmed results between the method detection limit and the LOQ, if reported at all, must be qualified as estimated concentrations. The laboratory's routine reporting limit (RL) is equal to the LOQ, and higher reporting limits may be used to satisfy special project requirements.

Special project RLs can be lower than the lab's standard LOQ if there is a written agreement with the client that poorer precision and bias are acceptable. The client must be informed in writing (e.g., confirmation of communication, letter of agreement, QAPP or report narrative) of the likelihood of less accurate quantitation, increased probability of false positive and false negative results, potential method compliance problems, and/or potential misidentification at the lower concentration. The RL can never be below the method detection limit.

#### **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.

#### **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. If the laboratory is developing a method without a published source method, then more extensive validation is required. The laboratory must establish the bias and precision in each sample matrix of interest throughout the working range.

#### **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.

#### **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.

### **19.4 Permitting Departures from Documented Procedure**

Each laboratory must have a procedure that defines the process, documentation, and level of authorization required to permit departures from documented procedures.

Where a departure from a documented SOP, test method, or policy is determined to be necessary, or unavoidable, the departure shall be documented and be authorized by the appropriate level of management, which is defined in the policy. In some instances, it is appropriate to inform the client before permitting a departure. Any such occurrence is documented in the cover letter and/or project narrative.

## **20.0 Equipment (and Calibration)**

### **20.1 Equipment Operation**

TestAmerica is committed to routinely updating and automating instrumentation. TestAmerica facilities maintain state of the art instrumentation to perform the analyses within the QC specifications of the test methods. Each laboratory shall maintain an equipment list that must include the following information:

- ❖ Date Installed or year placed in service
- ❖ Manufacturer's Name, Model Number, Serial Number
- ❖ Condition when Received

All equipment is subject to rigorous checks upon its receipt, upgrade, or modification to establish that the equipment meets with the selectivity, accuracy, and precision required by the test method for which it is to be used. All manufacturer's operations and maintenance manuals are kept up to date and accessible for the use of the equipment operator. Documentation of equipment usage is maintained using analytical run and maintenance logbooks or the electronic versions of said documents.

### **20.2 Equipment Maintenance**

Each laboratory must employ a system of preventative maintenance in order to ensure system up time, minimize corrective maintenance costs and ensure data validity. All routine maintenance is performed as recommended by the manufacturer and may be performed by an analyst, instrument specialist or outside technician. Maintenance logbooks or electronic records are kept on all major pieces of equipment in which both routine and non-routine maintenance is recorded. Notation of the date and maintenance activity is recorded each time service procedures are performed. The return to analytical control following instrument repair is documented. Maintenance logbooks or electronic records are retained as QA records.

Maintenance contracts are held on specific pieces of equipment where outside service is efficient, cost-effective, and necessary for effective operation of the laboratory.

### **20.3 Equipment Verification and Calibration**

All equipment shall be tested upon receipt to establish its ability to meet the QC guidelines contained in the test method for which the instrumentation is to be used. This testing shall be documented. Once an instrument is placed in routine service, ongoing instrument calibration is demonstrated at the appropriate frequency as defined in the test method. Refer to the Corporate Policy CA-T-P-002, *Selection of Calibration Points*, for guidance on using calibration data. Any instrument that is deemed to be malfunctioning is clearly marked and taken out of service. When the instrument is brought back into control, acceptable performance is documented.

### **20.4 Calibration**

Each laboratory must define calibration protocols in their facility-specific SOPs. Refer to the Corporate SOP CA-Q-S-005, *Calibration Curves*, for guidance on the calibration curve models used at TestAmerica and the basic formulae and calculations associated with them.

## **20.5 Glassware Cleaning**

Each laboratory must define glassware cleaning procedures in their facility SOPs.

## **20.6 Data Integrity and Security**

This section details those procedures that are relevant to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data.

### **Security and Traceability**

Access to computer systems that collect, analyze, and process raw instrumental data, and those that manage and report data must be both controlled and recorded. There are various systems at TestAmerica to which this applies, which include the Laboratory Information Management System (LIMS), as well as specific systems such as chromatography data systems.

Control of the system is accomplished through limitation of access to the system by users with the education, training and experience to perform the task knowledgeably and accurately. System users are granted privileges that are commensurate with their experience and responsibilities.

Computer access is tracked by using unique login names and passwords for all employees that have access to the computer system. "General" or "multi-user" account access to computer systems that collect, analyze and process raw instrumental data, and those that manage and report data shall not be permitted unless approved by laboratory management. Entries and changes are documented with the identity of the individual making the entry, and the time and date. Where a computer system is processing raw instrumental data, the instrument identification number is recorded. Many of these systems have the capability of maintaining audit trails to track entries and changes to the data. This function is activated on any computer system that has that capability.

TestAmerica requires that all sensitive computer systems, defined as LIMS servers and other servers of critical importance, be locked in a secured room. Access must be limited only to employees who need physical access to those systems. This room must also provide climate control within the parameters provided by the vendor of the secured equipment.

### **Verification**

All commercially obtained software shall be verified prior to use and after version upgrade. Verification involves assessing whether the computer system accurately performs its intended function. Verification generally is accomplished by comparing the output of the program with the output of the raw data manually processed, or processed by the software being replaced. The records of the verification are required to contain the following information: software vendor, name of product, version, comparison of program output and manual output, raw data used to verify the program, date, and name of the individual performing the verification. Records of verification are retained as records with IT personnel.

### **Validation**

Software validation involves documentation of specifications and coding as well as verification of results. Software validation is performed on all in-house programs. Records of validation include original specifications, identity of code, printout of code, software name, software

version, name of individual writing the code, comparison of program output with specifications, and verification records as specified above. Records of validation are retained as records with IT personnel.

### **Auditing**

The QA Departments system audits may include review of the control, security, and tracking of IT systems and software.

### **Version Control**

The laboratory shall maintain copies of outdated versions of software and associated manuals for all software in use at the laboratory for a period of five years from its retirement date. The associated hardware, required to operate the software, must also be retained for the same time period.

## **21.0 Measurement Traceability**

### **21.1 General**

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. 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.

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.

Laboratory DI and RO water systems have documented preventative maintenance schedules and the conductivity of the water is recorded on each day of use.

### **21.2 Reference Standards Traceability**

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 standards should be purchased 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 documentation of standard purity is archived, and references the Standard Identification Number.

All efforts are made to purchase standards that are  $\geq 97.0\%$  purity or as prescribed by the methods. If this is not possible, the purity is used in performing standards calculations.

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 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 is used as the second source confirmation.

### **21.3      Reagents**

Reagents are, in general, required to be analytical reagent grade unless otherwise specified in method SOPs. Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented.

## **22.0      Sampling**

### **22.1      Sampling Plans**

Sample representativeness and integrity are the foundations upon which meaningful analytical results rely. Where documented and approved SAPs and/or QAPPs are in place, they must be made available to the laboratory before sample receipt, and approved by laboratory management before sample receipt.

## **23.0      Handling of Samples**

### **23.1      General**

Chain of Custody (COC) can be established either when bottles are sent to the field, or at the time of sampling. TestAmerica can provide all of 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.

Samples are received at the laboratory by a designated sample custodian and a unique Laboratory Project Identification Number is assigned. The following information is recorded for each sample shipment: Client/Project Name, Date and Time of Laboratory Receipt, Laboratory Project Number, and Signature or Initials of the personnel receiving the cooler and making the entries.

Upon inspection of the cooler and custody seals, the sample custodian opens and inspects the contents of the cooler, and records the cooler temperature. If the cooler arrival temperature exceeds the required or method specified temperature range; sample receipt is considered "compromised" and the procedure described in Section 23.2 is followed. All documents are immediately inspected to assure agreement between the test samples received and the COC.

Any non-conformance, irregularity, or compromised sample receipt as described in Section 23.2 must be documented 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 permanent project record.

Samples that are being tested at another TestAmerica laboratory or by an external subcontractor shall be appropriately packaged and sent out under COC.

Following sample labeling as described in Section 23.3, the sample is placed in storage. Sample storage is required to be access-controlled. All samples are stored according to the requirements outlined in the test method and in a manner such that they are not subject to cross contamination or contamination from their environment. Unless specified by method or state regulation, a tolerance range of 0-6°C is used. Sample storage temperatures are monitored daily.

### **23.2 Sample Acceptance Policy**

Each laboratory shall maintain a sample acceptance policy that describes compromised sample receipt. Samples shall be 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 preservative.
- ❖ 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.
- ❖ Apparent tampering with cooler and/or samples.
- ❖ Headspace in volatiles samples.
- ❖ Seepage of extraneous water or materials into samples.
- ❖ Inadequate sample volume.
- ❖ Illegible, impermanent, or non-unique sample labeling.

When "compromised" samples are received, it must be documented by the laboratory and the client must be contacted for instructions. If the client decides to proceed with the analysis, the project report shall clearly indicate any of the above conditions and the resolution.

### **23.3 Sample Identification and Traceability**

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 sample identification label.

All unused portions of samples are returned to the secure sample control area. Where required by the project, empty sample containers are also retained.

#### **23.4 Sub-sampling**

Sub-sampling procedures must be referenced in each facility's QAM and documented in their SOPs.

#### **23.5 Sample Preparation**

Sample preparation procedures must be referenced in each facility's QAM and documented in their SOPs.

#### **23.6 Sample Disposal**

Each facility shall have an SOP describing sample retention and disposal procedures. Samples should be retained in TestAmerica storage facilities 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. Samples may be returned to the client per written request. 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.

Samples shall be disposed of in accordance with federal, state and local regulations. Each facility must have an SOP detailing the disposal of samples, digestates, and extracts. All laboratories shall remove or deface sample labels prior to disposal unless this is accomplished through the disposal method (e.g., samples are incinerated).

### **24.0 Assuring the Quality of Test Results**

#### **24.1 Control Samples**

Control samples are analyzed with each batch of samples to monitor laboratory performance in terms of accuracy, precision, sensitivity, selectivity, and interferences. Each regulatory program and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch. Control samples must be uniquely identified and correlated to unique batches. There are also a number of QC sample types that monitor field sampling accuracy, precision, representativeness, interferences, and the effect of the matrix on the method performed. Control sample types and typical frequency of their application are outlined in Table 24-1. Note that frequency and use of control samples vary with specific regulatory, methodology and project specific criteria. Table 24-1 does not define TestAmerica's approach to the application of QC samples for each regulatory program or test method.

**Table 24-1. Example of Control Samples**

<b>Laboratory QC Sample Type</b>	<b>Use</b>	<b>Required Frequency</b>
Laboratory Control Sample (LCS) (Laboratory Fortified Blank)	Measures accuracy of the method in a blank matrix	Generally 1 for each batch of samples; not to exceed 20 environmental samples.
Method Blank (MB)	Measures method contribution to any source of contamination	Generally it is 1 for each batch of samples; not to exceed 20 environmental samples.
Instrument Blank	Measures instrumental contribution to any source of contamination	As specified in test method
Reference Toxicant	Measure sensitivity of test organisms (Aquatic toxicology)	Annually
<b>Field QC Sample Type</b>	<b>Use</b>	<b>Typical Frequency</b>
Matrix Duplicate	Measures the effect of the site matrix on the precision of the method	Per 20 samples per matrix or per SAP/QAPP <sup>1</sup>
Matrix Spike	Measures the effect of the site matrix on the accuracy of the method	Per 20 samples per matrix or per SAP/QAPP
Matrix Spike Duplicate	Measures the effect of the site matrix on the precision of method	Per 20 samples per matrix or per SAP/QAPP <sup>1</sup>
Equipment Blank (Equipment Rinsate)	Measures field equipment contribution to any source of contamination	Per SAP/QAPP
Trip Blank	Measures shipping contribution to any source of contamination (Volatiles only)	Per Cooler
Field Blank	Measures the field environment contribution to any source of contamination	Per SAP/QAPP
Field Duplicate	Measures representativeness of the sampling and the effect of the site matrix on precision	Per SAP/QAPP

<sup>1</sup> Either an MSD or an MD is required per 20 samples per matrix or per SAP/QAPP.

## **24.2 Review / Verification Procedures**

The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review COC forms and input the sample information and required analyses into a computer LIMS. A secondary review of the transaction of the chain-of-custody forms and the inputted information is also performed by sample control personnel. The PMs perform final review of the COC forms and inputted information.

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

Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, PM, QA Manager, Technical Manager, or Supervisor for further investigation. Corrective action is initiated whenever necessary.

Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements.

All data, regardless of regulatory program or level of reporting, shall be subject to a thorough review which involves a primary, secondary, and completeness review process. All levels of the review must be documented.

Any anomalous results and/or non-conformances noted during the Primary Review are documented on a data review checklist (defined by each facility) communicated to the Supervisor and the PM for resolution. Resolution can require sample reanalysis, or it may require that data be reported with a qualification. Non-conformances are documented per Section 12.

#### **24.2.1 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 must train all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP (CA-Q-S-002) as the guideline.

### **24.3 Development of QC Criteria, Non-Specified in Method/Regulation**

Where a method or regulation does not specify acceptance and/or rejection criteria, the laboratory must develop a policy for doing so. The policy must address how the laboratory examines the data user's needs and the demonstrated sensitivity, accuracy and precision of the available test methods in determining appropriate QC criteria.

Data users often need the laboratory's best possible sensitivity, accuracy, and precision using a routinely offered test method, or are unsure of their objectives for the data. For routine test methods that are offered as part of TestAmerica's standard services, the laboratory bases the QC criteria on statistical information such as determination of sensitivity, historical accuracy and precision data, and method verification data. The method SOP includes QC criteria for ongoing demonstration that the established criteria are met (e.g., acceptable LCS accuracy ranges, precision requirements, method blank requirements, initial and continuing calibration criteria, etc.).

In some cases, a routine test method may be far more stringent than a specific data user's needs for a project. The laboratory may either use the routinely offered test method, or may opt to develop an alternate test method based on the data user's objectives for sensitivity, accuracy, and precision. In this case, it can be appropriate to base the QC criteria on the data user's objectives, and demonstrate through method verification and ongoing QC samples that these objectives are met.

For example, a client may require that the laboratory test for a single analyte with specific DQOs for sensitivity, accuracy, and precision as follows: Reporting Limit of 10 ppm, accuracy  $\pm 25\%$ , and RSD of less than 30%. The laboratory may opt to develop a method that meets these criteria and document the results of the Method Blanks, MDL study, and LCSs that the method satisfies those objectives. In this case, both the method and the embedded QC criteria have been based on the client's DQOs.

In some cases, the data user needs more stringent sensitivity, accuracy, and/or precision than the laboratory can provide using a routine test method. In this case, it is appropriate that the laboratory provide documentation of the sensitivity, accuracy, and precision obtainable to the data user and let the data user determine whether to use the best available method offered by the laboratory, or determine whether method development or further research is required.

## **25.0 Reporting Results**

### **25.1 Project Reports**

All TestAmerica Project Reports that are generated under NELAC requirements must contain the content as described below. This criteria applies to all Project Reports.

### **25.2 Project Report Content**

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. At a minimum, the standard laboratory report shall contain the following information:

- ❖ A report title (e.g. Analytical Report for Samples) with a “sample results” column header.
- ❖ Each report cover page printed on company letterhead, which includes the laboratory name, address and telephone number.
- ❖ 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.
- ❖ A copy of the chain of custody (COC).
- ❖ The name and address of client and a project name/number, if applicable.
- ❖ Client project manager or other contact
- ❖ Description and unambiguous identification of the tested sample(s) including the client identification code.
- ❖ 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.
- ❖ Date reported or date of revision, if applicable.
- ❖ Method of analysis including method code (EPA, Standard Methods, etc).
- ❖ Practical quantitation limits or reporting limit.
- ❖ Method detection limits (if requested)
- ❖ Definition of Data qualifiers and reporting acronyms (e.g., ND).
- ❖ Sample results.
- ❖ QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits (if requested)
- ❖ Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets.
- ❖ A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.
- ❖ A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- ❖ A statement that the report shall not be reproduced except in full, without prior express written approval by the Laboratory Director or PM.
- ❖ 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 Laboratory Director.
- ❖ 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.
- ❖ The laboratory includes a cover letter.
- ❖ 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.
- ❖ When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.
- ❖ Appropriate laboratory certification number for the state of origin of the sample, if applicable.

- ❖ 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.
- ❖ 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.

### **25.3 Electronic Data Deliverables**

Electronic Data Deliverables (EDD) are routinely offered as part of TestAmerica's services. TestAmerica offers a variety of EDD formats including Environmental Restoration Information Management System (ERPIMS), New Agency Standard (NAS), SEDD, Format A, 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 outlined in Section 7. Once the facility has committed to providing diskettes in a specific format, the coding of the format may need to be performed. This coding is documented and validated. The validation of the code is retained as a QC record.

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 Project Report Format**

TestAmerica offers a wide range of project reporting formats, including EDDs, short report formats, and complete data deliverable packages modeled on the Contract Laboratory Protocol (CLP) guidelines. More information on the range of project reports available can be obtained by contacting any TestAmerica laboratory. Regardless of the level of reporting, all projects must undergo the levels of review as described in Section 24.2.

**Appendix 1. List of TestAmerica Corporate Policies & SOPs**

<b>Document No.</b>	<b>Title</b>
CA-Q-S-005	Calibration Curves (General)
CW-Q-S-001	Corporate Document Control and Archives
CA-Q-S-006	Detection Limits
CA-I-P-002	Electronic Reporting and Signature Policy
CA-L-P-001	Ethics Policy
CA-Q-S-002	Manual Integrations
CA-Q-S-008	Quality Systems Management Review
CA-T-P-002	Selection of Calibration Points
CA-Q-S-001	Solvent & Acid Lot Testing & Approval
CA-L-S-002	Subcontracting Procedures



ARCADIS

**Appendix C**

Selected Boring Logs



# THE H. C. NUTTING COMPANY

GEOTECHNICAL AND TESTING ENGINEERS

SINCE 1921

912 MORRIS STREET  
CHARLESTON, W. VA 25301  
304-344-0821

4128 AIRPORT ROAD  
P.O. BOX C  
CINCINNATI, OHIO 45226  
513-321-5816

BOX NUMBER 11  
HIGHLAND HEIGHTS, KY 41076  
606-261-2043

"AS A MUTUAL PROTECTION TO CLIENTS, THE PUBLIC, AND OURSELVES, ALL REPORTS ARE SUBMITTED AS THE CONFIDENTIAL PROPERTY OF CLIENTS, AND AUTHORIZATION FOR PUBLICATION OF STATEMENTS, CONCLUSIONS, OR EXTRACTS FROM OR REGARDING OUR REPORTS IS RESERVED PENDING OUR WRITTEN APPROVAL."

## FIELD TEST BORING REPORT

10-18-82 vf  
Page 1 of 2

CLIENT Geraghty & Miller, Inc. ORDER No. 10191.001

PROJECT GMC, Harrison Radiator Division, Monitoring Wells, HOLE No. GM-15  
Moraine, Ohio

LOCATION As shown on well location plan

DRILLER Willard Martin DRILL No. 36 DATE STARTED 9-13-82  
Top of ex-well guard pipe adj. to GM-5; with

ELEVATION REFERENCE cap off (see well location plan) Elev. 731.48 DATE COMPLETED 9-13-82

CASING: DIAMETER 3.5" I. D. Hollow Stem Auger HAMMER WT. FALL

SAMPLER: DIAMETER & TYPE 2" O. D. Split Spoon HAMMER WT. 140# FALL 30"

DEPTH TO WATER: IMMEDIATE 35.0' UPON COMPLETION

DEPTH TO WATER        DAYS AFTER COMPLETION        WATER USED IN DRILLING       

ELEVATION	DEPTH	DESCRIPTION OF MATERIALS	SAMPLE No.	SAMPLE DEPTH	TYPE OF SAMPLE	BLOWS PER 6" ON SAMPLER <small>or % Core Rec.</small>	Recor.
724.6	0'	Silt, topsoil, roots, some fine sand, brown	1	0-1.5	SS	6-14-15	18"
		Sand and gravel, poorly sorted, brown sand is fine to medium, gravel is coarse	2	5-6.5	SS	10-13-9	18"
		Sand and gravel, poorly sorted, brown, sand is fine to medium, gravel is fine to coarse, 1" of sand was damp	3	10-11.5	SS	13-25-25	10"
		Gravel and sand, poorly sorted, very slight oil odor	4	15-16.5	SS	16-30-4	12"
		Sand and gravel, poorly sorted, oil, odor, some damp spots	5	20-21.5	SS	15-26-27	15"
		Sand and gravel, poorly sorted, no odor, iron stains	6	25-26	SS	12-60	10"
		Sand and gravel, fine to coarse, brown, iron stains, moist to wet in top of spoon	7	30-31.5	SS	16-32-48	18"
		Encountered water at 35.0'					

A-33

Respectfully submitted,  
THE H. C. NUTTING CO.

Samples recovered from this test boring are available for inspection, which is strongly recommended. The company assumes no responsibility for interpretations made by others of load bearing, stability, excavating or other physical

PROJECT GMC, Harrison Radiator Division, Monitoring Wells, HOLE No. GM-15  
Moraine, Ohio

ELEVATION	DEPTH	DESCRIPTION OF MATERIALS	SAMPLE No.	SAMPLE DEPTH	TYPE OF SAMPLE	BLOWS PER 6" DN SAMPLER BY % CORE REC.	R
		Sand, medium to coarse and gravel, little silt and fine sand, wet, brown	8	35-36.5	SS	12-50-26	1
		Sand, medium to coarse and gravel, little silt and fine sand, wet, brown	9	40-41.5	SS	21-28-38	
		Sand, medium to coarse some gravel, little silt and fine sand, wet, brown	10	45-46.5	SS	16-31-40	1
		Gravel, some fine to coarse sand, trace silt, brown, wet	11	50-51.5	SS	15-18-36	
		Sand, fine to coarse, some gravel, little silt, brown, wet	12	55-56.5	SS	21-28-4	14
		Sand, fine to coarse, some gravel, little silt, brown, wet, silt, some fine sand and gravel - pebble, gray	13	60-61	SS	41-60	
		Silt, some fine to coarse sand and gravel, gray, moist, little clay	14	65-66	SS	21-62	12
		Silt and fine sand, little medium to coarse sand and gravel and clay, gray, moist	15	70-71.5	SS	12-21-33	18
		Sand, fine, some silt, gray, wet	16	75-76	SS	38-55	16
		Sand, fine, some silt, gray, wet	17	80-81	SS	47-62	12
		Sand, fine, some silt, gray, wet	18	85-86	SS	37-56	
		Sand, fine to coarse, little gravel and silt, brown, wet	19	90-91	SS	36-58	
		Sand, fine to coarse and gravel, trace silt, brown, wet	20	95-96	SS	50-62	
		No sample BORING COMPLETED	21	100-101.5	SS	23-25-37	
		REMARKS: 1. Logged by G.F. Schank (G&M) 2. Monitoring well set at 100.0' (see well detail table)					



**THE H. C. NUTTING COMPANY**  
 GEOTECHNICAL AND TESTING ENGINEERS  
 SINCE 192

912 MORRIS STREET  
 CHARLESTON, W. VA 25301  
 304-344-0821

4120 AIRPORT ROAD  
 P.O. BOX C  
 CINCINNATI, OHIO 45226  
 513-321-5816

BOX NUMBER 11  
 HIGHLAND HEIGHTS, KY 41076  
 606-261-2043

"AS A MUTUAL PROTECTION TO CLIENTS, THE PUBLIC, AND OURSELVES, ALL REPORTS ARE SUBMITTED AS THE CONFIDENTIAL PROPERTY OF CLIENTS, AND AUTHORIZATION FOR PUBLICATION OF STATEMENTS, CONCLUSIONS, OR EXTRACTS FROM OR REGARDING OUR REPORTS IS RESERVED PENDING OUR WRITTEN APPROVAL."

**FIELD TEST BORING REPORT**

10-18-82 vf

CLIENT Geraghty & Miller, Inc. ORDER No. 10191.001  
 PROJECT GMC, Harrison Radiator Division, Monitoring Wells HOLE No. GM-16  
Moraine, Ohio  
 LOCATION As shown on well location plan  
 DRILLER Willard Martin DRILL No. 36 DATE STARTED 9-14-82  
Top of ex-well guard pipe adj. to GM-5; with  
 ELEVATION REFERENCE cap off (see well location plan) Elev. 731.48 DATE COMPLETED 9-14-82  
 CASING: DIAMETER 3.5" I. D. Hollow Stem Auger HAMMER WT. FALL  
 SAMPLER: DIAMETER & TYPE None HAMMER WT. FALL  
 DEPTH TO WATER: IMMEDIATE UPON COMPLETION  
 DEPTH TO WATER DAYS AFTER COMPLETION WATER USED IN DRILLING

ELEVATION	DEPTH	DESCRIPTION OF MATERIALS	SAMPLE No.	SAMPLE DEPTH	TYPE OF SAMPLE	BLOWS PER 6" ON SAMPLER or % Core Rec.	Re:
724.6'	0'						
	60.0'	60.0' No samples, see GM-15					
664.6	60.0'	BORING COMPLETED					
		REMARKS:					
		1. Monitoring well set at 58.0' (see well detail table)					

Respectfully submitted,  
**THE H. C. NUTTING CO.**

Samples recovered from this test boring are available for inspection, which is strongly recommended. The company assumes no responsibility for interpretations made by others of load bearing, stability, excavating or other physical

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0	N/A	12	0.0				GP	GRAVEL Poorly graded, dry	
2	N/A	12	0.0				SW	SAND AND GRAVEL Brown, medium-fine, gravel (20%), little silt, dry	
4	N/A	12	0.0				SW	Same as above	
6	N/A	12	0.0				SW	Same as above	
8	N/A	24	0.0				SW	Same as above	
10	N/A	24	0.0				SW	Same as above	
12	N/A	24	0.0				SW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
14	N/A	24	0.0				GP	GRAVEL Poorly graded, dry	
16	N/A	24	0.0				SW	SAND & GRAVEL Brown, medium-fine sand, gravel (10%), little silt, dry	
18	N/A	8	0.0				SW	Same as above, gravel (30%)	
20	N/A	8	0.0				SW	Same as above	
22	N/A	8	0.0				SW	Same as above	
24	N/A	8	0.0				SW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 2 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
26	N/A	20	0.0				SW	Same as above, wet at 27'	▼
28	N/A	20	0.0				GP	GRAVEL Poorly graded, wet	
30	N/A	20	0.0				GW	GRAVEL Brown, coarse sand (40%), wet  Water sample collected at 30' for site specific VOCs.	
32	N/A	20	0.0				GW	Same as above	
34	N/A	20	0.0				GW	Same as above, medium-coarse sand (30%)	
36	N/A	24	1.3				GW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 3 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
38	N/A	24	1.3				SW	SAND & GRAVEL Brown, medium-coarse sand (50%), fine gravel, wet	
40	N/A	24	1.5				SW	Same as above	
42	N/A	24	1.4				SW	Same as above	
44	N/A	24	1.4				SW	Same as above	
								----- Water sample collected at 45' for site specific VOCs.	
46	N/A	20	1.4				SW	Same as above, gravel (20%)	
48	N/A	20	0.0				SW	Same as above	
50									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 4 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
52	N/A	20	0.0				SW	Same as above, fine gravel (50%)	
54	N/A	20	0.0				SW	Same as above, gravel (10%)	
56	N/A	24	0.0				SW	SAND Brown, medium-coarse, wet	
58	N/A	24	0.0				SP	SAND Brown, medium, little silt, poorly graded, saturated	
60	N/A	24	0.0				CL	SILTY CLAY Gray, gravel (10-20%), stiff-very stiff, low plasticity, 4" gravel layer above silty clay  Water sample collected at 60' for site specific VOCs.	
62	N/A	24	0.0				CL	Same as above, gravel (10%)	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 5 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
64	N/A	24	0.0				CL	Same as above	
66	N/A	12	0.0				CL	Same as above, gravel (20%)	
68								End of boring	
70									
72									
74									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 6 of 6

Drilling Co.: Prosonic Corporation     Geologist: T. Fortner     Begin Drilling: 2/23/06 @ 1650  
 Driller: J. Sigler     Total Depth: 67     End Drilling: 2/24/06 @ 1126  
 Drilling Method: Rotosonic     Surface Elev.: 727.03     Converted to Well: Y Well I.D.: GM-47  
 Drilling Fluid: Water     North Coord.: 1312.270     East Coord.: 4776.411

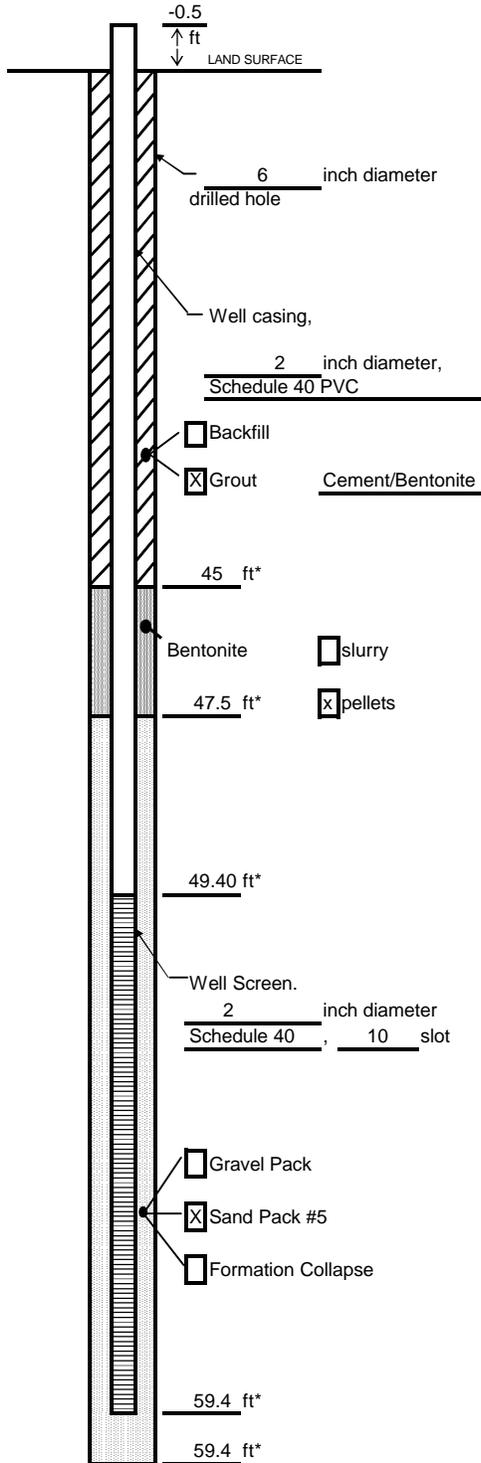
Remarks: \_\_\_\_\_

Project No.: OH000294.0008.00002     Datum: TOC Elev. 725.75     Filename: February 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Project General Motors Corporation Well GM-47

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

727.03 feet  Surveyed

Estimated

Installation Date(s) 2/24/2006

Drilling Method Rotosonic

Drilling Contractor Prosonic Corporation

Drilling Fluid Water

Development Technique(s) and Date(s)

Submersible pump 2/25/06

Fluid Loss During Drilling NA gallons

Water Removed During Development 600 gallons

Static Depth to Water 21.91 feet below M.P.

Pumping Depth to Water 50-59.5 feet below M.P.

Pumping Duration 4.00 hours

Yield 3 gpm Date 2/25/06

Specific Capacity N/A gpm/ft

Well Purpose Monitoring Well

Remarks TOC Elevation = 726.75

Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.

\* Depth Below Land Surface

Prepared by J. Manzo

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0								See GM-47 for lithologic description from 0-40'	
2									
4									
6									
8									
10									
12									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 1

Drilling Co.: Boart Longyear    
 Geologist: T. Fortner    
 Begin Drilling: 4/24/06

Driller: B. Smith    
 Total Depth: 40    
 End Drilling: 4/24/06

Drilling Method: Rotosonic    
 Surface Elev.: 727.034    
 Converted to Well: Y Well I.D.: GM-50

Drilling Fluid: Water    
 North Coord.: 1311.64197    
 East Coord.: 4773.28499

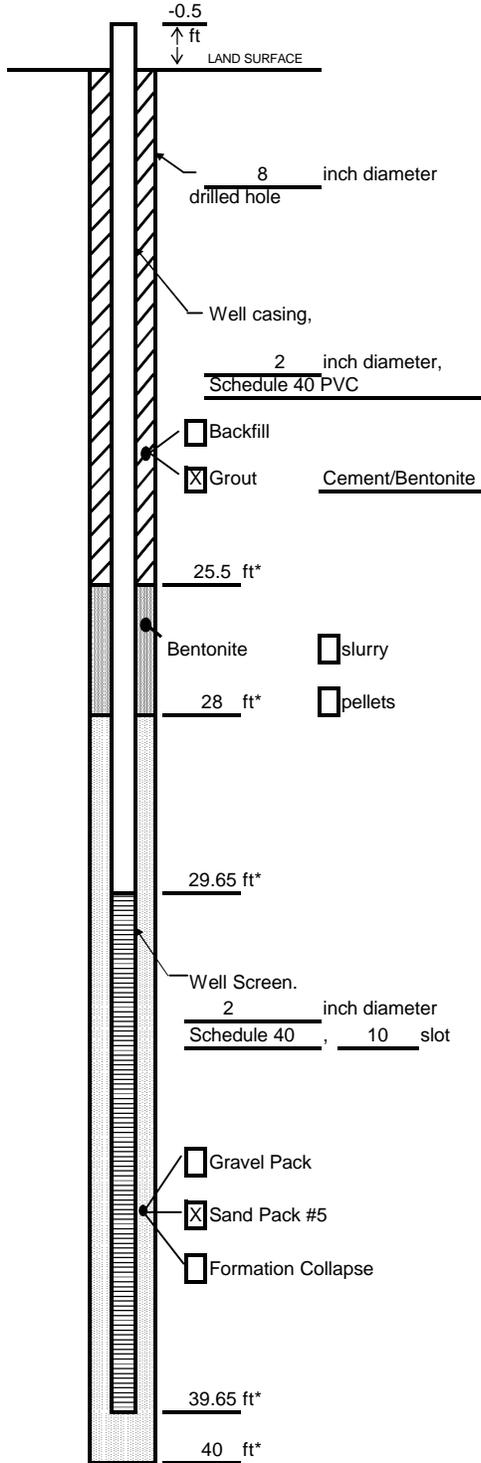
Remarks: Shallow pair to GM-47.

Project No.: OH000294.0008.00002    
 Datum: TOC Elev = 722.109    
 Filename: July 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Project General Motors Corporation Well GM-50

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

727.034 feet  Surveyed

Estimated

Installation Date(s) 4/24/2006

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)

Pumping - surge with pump 4/26/06

Fluid Loss During Drilling NA gallons

Water Removed During Development 200 gallons

Static Depth to Water 20.39 feet below M.P.

Pumping Depth to Water NM feet below M.P.

Pumping Duration 1.50 hours

Yield NM gpm Date 4/26/06

Specific Capacity NM gpm/ft

Well Purpose Monitoring Well

Remarks TOC Elevation = 726.555

Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.

\* Depth Below Land Surface

Prepared by J. Manzo

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0								See GM-54 for lithologic description from 0-33'	
2									
4									
6									
8									
10									
12									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Drilling Co.: Boart Longyear

Geologist: T. Fortner

Begin Drilling: 7/25/06

Driller: M. Osterberg

Total Depth: 33

End Drilling: 7/25/06

Drilling Method: Rotosonic

Surface Elev.: 730.530

Converted to Well: Y Well I.D.: GM-53

Drilling Fluid: Water

North Coord.: 2999.31791

East Coord.: 6823.94469

Remarks: Shallow pair to GM-54.

Project No.: OH000294.0008.00002

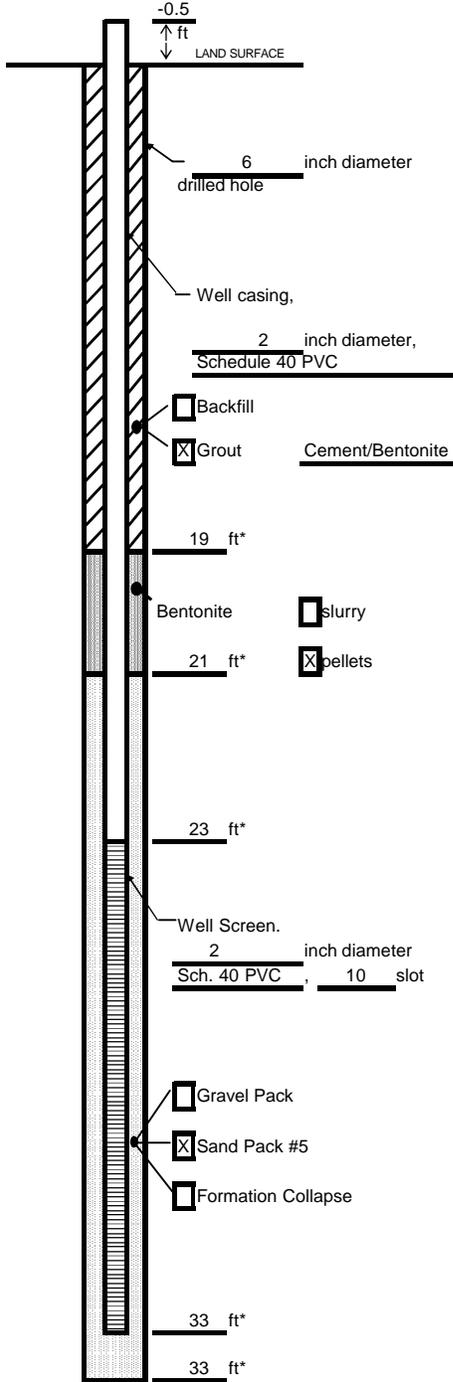
Datum: TOC Elev 730.353

Filename: July 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.  
Unless Otherwise Noted.  
\* Depth Below Land Surface

Project General Motors Corporation Well GM-53

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:  
730.530 feet  Surveyed  Estimated

Installation Date(s) 7/28/2006

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)  
Pumping - surge with pump 7/30/06

Fluid Loss During Drilling -100 gallons

Water Removed During Development 120 gallons

Static Depth to Water 22.87 feet below M.P.

Pumping Depth to Water NM feet below M.P.

Pumping Duration 0.70 hours

Yield 3 gpm Date 7/30/06

Specific Capacity NM gpm/ft

Well Purpose Monitoring well

Remarks TOC Elevation = 730.353

Time 1040, 1043, 1046, 1049, 1052

pH 6.39, 6.49, 6.50, 6.49, 6.50

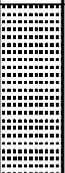
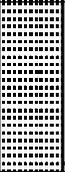
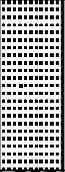
Conductivity 1.74, 1.66, 1.66, 1.66, 1.66

Turbidity 0, 0, 0, 0, 0 (clear-nonturbid)

Temperature 19.3, 18.8, 17.8, 17.9, 17.8

Prepared by T. Fortner

### General Motors Corporation Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0	N/A	0	N/A					No recovery	
2									
4									
6	N/A	24	3.3				SW	SAND Brown, medium-coarse, medium gravel (20-30%), well graded, dry	
8	N/A	24	8.3				SW	Same as above	
10	N/A	24	1.4				SW	Same as above	
12	N/A	24	0.7				GW	GRAVEL Brown, medium-coarse, medium-coarse sand (30%), well graded, dry	
								GRAVEL Brown, medium-coarse, medium-coarse sand (30%), well graded, dry	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

### General Motors Corporation Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
14	N/A	24	1.6				SW	SAND Brown, medium-coarse, fine-medium gravel (40%), well graded, dry	
16	N/A	24	1.0				GW	GRAVEL Brown, medium-coarse, medium-coarse sand (30%), well graded, dry	
18	N/A	24	2.7				GW	Same as above	
20	N/A	24	1.5				GW	Same as above	
22	N/A	24	1.1				GW	Same as above, wet at 22'	
24	N/A	24	1.2				GW	Same as above	
								Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 2 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

### General Motors Corporation Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
26	N/A	24	1.5				SP	SAND Brown, medium grained, fine gravel (5-10%), poorly graded, wet	
28	N/A	24	0.8				SP	Same as above	
30	N/A	24	0.1				SP	Same as above	
32	N/A	24	0.4				GW	GRAVEL Brown, fine-medium, medium-coarse sand (30%), well graded, wet	
34	N/A	24	0.1				CL	SANDY CLAY Brown, stiff, low plasticity with trace gravel, moist	
36	N/A	24	0.5				CL	SILTY CLAY Gray, stiff, brittle, low plasticity with ~5% fine gravel, dry	
								Same as above	
	N/A	24	0.3				CL	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 3 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (16 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
38	N/A	24	0.5				CL	Same as above	
40	N/A	24	0.4				CL	Same as above	
42	N/A	24	0.4				CL	Same as above	
44	N/A	24	0.0				CL	Same as above	
46	N/A	24	0.0				CL	Same as above	
48	N/A	24	0.0				CL	Same as above	
50	N/A	24	0.0				CL	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 4 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
52	N/A	24	0.0				CL	Same as above	
54	N/A	24	0.0				CH	SILTY CLAY Gray with some coarse sand, soft, high plasticity, moist-wet	
56	N/A	24	0.0				CL	SILTY CLAY Gray, stiff, brittle, low plasticity, medium gravel (5%), dry	
58	N/A	24	0.0				CL	Same as above	
60	N/A	24	0.0				CL	Same as above	
62	N/A	24	0.0				CL	Same as above	

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 5 of 10

Drilling Co.: Boart Longyear

Geologist: T. Fortner

Begin Drilling: 7/11/06 @ 1550

Driller: M. Osterberg

Total Depth: 115

End Drilling: 7/12/06 @ 1300

Drilling Method: Rotosonic

Surface Elev.: 730.513

Converted to Well: Y Well I.D.: GM-54

Drilling Fluid: Water

North Coord.: 2995.14271

East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002

Datum: TOC Elev = 730.287

Filename: July 2006

### General Motors Corporation Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
64	N/A	24	0.3				CL	Same as above	
							SW	SAND Brown, medium-coarse, coarse gravel (5%), well graded, wet	
66	N/A	24	0.4				SW	Same as above	
68	N/A	24	0.1				SW	Same as above	
70	N/A	24	0.2				GW	GRAVEL Brown, medium-coarse, medium coarse sand (40%), wet	
72	N/A	24	0.0				SM	SILTY SAND Brown, fine, wet	
								Same as above	
74	N/A	24	0.5				SM	Same as above	
	N/A	24	0.0				SM	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 6 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
76	N/A	24	0.1				SP	SAND Brown, fine-medium, poorly graded, wet	
78	N/A	24	0.1				SP	Same as above	
80	N/A	24	0.5				SP	Same as above	
82	N/A	24	0.1				SP	Same as above	
84	N/A	24	14.7				SP	Same as above  (OVA readings from 85-103' were performed during intense rainfall; humidity levels may have produced instrument drift while screening.)	
86	N/A	24	16.8				SW	SAND Brown, fine-coarse, medium-coarse gravel (40%), well graded, wet	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 7 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
88	N/A	24	8.6				SW	Same as above	
90	N/A	24	4.2				SW	Same as above	
92	N/A	24	10.7				SW	Same as above	
94	N/A	24	6.6				SW	Same as above	
96	N/A	24	18.8				SW	SAND Brown, medium-coarse cobbles (20%), well graded, wet	
98	N/A	24	31.1				SW	SAND Brown, fine-medium, fine-medium gravel (40%), some silt, well graded, wet	
100									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 8 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
102	N/A	24	39.2				SW	Same as above	
104	N/A	24	0.0				SW	Same as above	
106	N/A	24	0.0				GM	SAND/GRAVEL/SILT Brown, fine-coarse sand (50%), fine-medium gravel (30%), silt (20%), well graded, wet	
108	N/A	24	0.0				SW	SAND Brown, medium-coarse, medium gravel (10%), well graded, wet	
110	N/A	24	0.0				SW	Same as above	
112	N/A	24	0.0				SW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 9 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 7/11/06 @ 1550  
 Driller: M. Osterberg     Total Depth: 115     End Drilling: 7/12/06 @ 1300  
 Drilling Method: Rotosonic     Surface Elev.: 730.513     Converted to Well: Y Well I.D.: GM-54  
 Drilling Fluid: Water     North Coord.: 2995.14271     East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'  
 Project No.: OH000294.0008.00002     Datum: TOC Elev = 730.287     Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
114	N/A	24	0.0				SW	Same as above	
116								End of boring	
118									
120									
122									
124									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 10 of 10

Drilling Co.: Boart Longyear

Geologist: T. Fortner

Begin Drilling: 7/11/06 @ 1550

Driller: M. Osterberg

Total Depth: 115

End Drilling: 7/12/06 @ 1300

Drilling Method: Rotosonic

Surface Elev.: 730.513

Converted to Well: Y Well I.D.: GM-54

Drilling Fluid: Water

North Coord.: 2995.14271

East Coord.: 6817.71652

Remarks: Water samples 70-75'; 80-85'; 95-100'; 110-115'

Project No.: OH000294.0008.00002

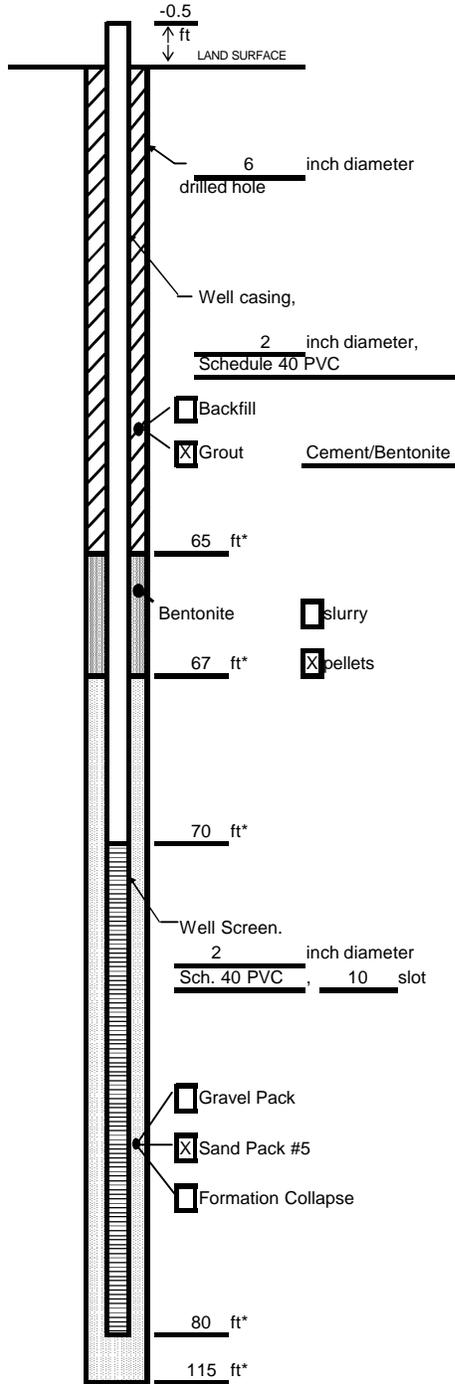
Datum: TOC Elev = 730.287

Filename: July 2006

ARCADIS

# Well Construction Log

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.  
\* Depth Below Land Surface

Project General Motors Corporation Well GM-54

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:  
730.513 feet  Surveyed  
 Estimated

Installation Date(s) 7/25/2006

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)  
Pumping - surge with pump 7/30/06

Fluid Loss During Drilling -300 gallons

Water Removed During Development 320 gallons

Static Depth to Water 21.68 feet below M.P.

Pumping Depth to Water NM feet below M.P.

Pumping Duration 1.20 hours

Yield 4 gpm Date 7/30/06

Specific Capacity NM gpm/ft

Well Purpose Monitoring well - Well B

Remarks TOC Elevation = 730.287

Time 1110, 1113, 1116, 1119, 1122

pH 6.65, 6.44, 6.46, 6.45, 6.45

Conductivity 1.28, 1.27, 1.27, 1.27, 1.27

Turbidity 0, 0, 0, 0, 0 (clear-nonturbid)

Temperature 19.0, 18.8, 18.7, 18.7, 18.6

Prepared by T. Fortner

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0	N/A	0	N/A					No recovery	
2									
4									
6									
8	N/A	24	0.0				SW	SAND Brown, medium-coarse, fine-medium gravel (30%), moist	
10	N/A	24	0.0				SW	Same as above, dry	
12	N/A	24	0.0				SW	Same as above	
							SW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
14	N/A	24	0.0			[Dotted Pattern]	SW	Same as above	
16	N/A	24	0.0			[Dotted Pattern]	SW	Same as above	
18	N/A	24	0.0			[Dotted Pattern]	SW	Same as above	
20	N/A	24	0.0			[Dotted Pattern]	SW	Same as above	
22	N/A	24	0.0			[Dotted Pattern]	SW	Same as above	
24	N/A	24	0.0			[Dotted Pattern]	SW	Same as above, wet	▼
24						[Dotted Pattern]	SW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 2 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

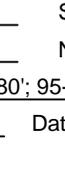
Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
26	N/A	24	0.0				SW	Same as above	
28	N/A	24	0.0				GW	GRAVEL Brown, fine-medium, medium-coarse sand (30%), well graded, wet	
30	N/A	24	0.0				GW	Same as above	
30	N/A	24	0.0				CL	SANDY CLAY Brown, stiff, moist, medium plasticity, moist	
32	N/A	24	0.0				CL	SILTY CLAY Gray, fine gravel (10%), stiff, dry, low-medium plasticity, moist	
34	N/A	24	0.0				CL	Same as above	
36	N/A	24	1.8				CL	Same as above, fine-medium gravel (40-50%)	
36	N/A	24	1.8				CL	Same as above	
36	N/A	24	2.9				CL	Same as above, very stiff, fine gravel (10%)	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 3 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
38	N/A	24	10.4				CL	Same as above	
40	N/A	24	16.1				CL	Same as above, fine-medium gravel (10%)	
42	N/A	24	17.3				CL	Same as above	
44	N/A	24	15.2				CL	SANDY CLAY Gray, fine sand, medium plasticity, soft, moist, 6 inches fine sand stringer, fine gravel (5%)	
46	N/A	24	14.6				CL	Same as above, 6 inches fine sand stringer, fine gravel (5%)	
48	N/A	24	16.0				CL	Same as above	
50	N/A	24	16.0				CL	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 4 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648  
 Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630  
 Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58  
 Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
52	N/A	24	17.0				CL	SILTY CLAY Gray, soft, medium plasticity, fine gravel 5%, dry	
							CL	Same as above	
54	N/A	24	17.1				CL	Same as above	
							SP	SAND Gray, fine, poorly graded	
56	N/A	24	16.7				CL	SANDY CLAY Gray, fine-medium sand, stiff, low plasticity, dry	
							CL	Same as above	
58	N/A	24	17.2				CL	SILTY CLAY Dark gray, fine-medium gravel (5%), trace organic material, stiff, low plasticity, dry	
							CL	Same as above	
60	N/A	24	18.2				CL	SILTY CLAY Brown, soft, medium plasticity, moist-dry	
							CL	Same as above	
62	N/A	24	22.4				CL	SILTY CLAY Brown, soft, medium plasticity, moist-dry	
							CL	Same as above	

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 5 of 10

Drilling Co.: Boart Longyear

Geologist: T. Fortner

Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell

Total Depth: 115

End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic

Surface Elev.: 735.588

Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water

North Coord.: 3450.80045

East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009

Datum: TOC Elev=735.462

Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
64	N/A	24	17.5				CL	Same as above	
66	N/A	24	17.5				CL	SILTY CLAY Gray, very stiff, low-medium plasticity, fine gravel (5-10%), dry	
68	N/A	24	18.1				CL	Same as above	
70	N/A	24	15.5				CL	Same as above	
70	N/A	24	N/A				SP	SAND Brown, fine-medium, little silt, wet	
72	N/A	24	N/A					No recovery	
72	N/A	24	N/A					No recovery	
74	N/A	24	N/A					No recovery	
	N/A	24	0.0				GP	GRAVEL	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 6 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648  
 Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630  
 Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58  
 Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
76	N/A	24	0.0					Brown, fine-medium, coarse sand (10-20%), poorly graded, wet	
78	N/A	24	0.0				SP	SAND Brown, medium-coarse, poorly graded, wet	
80	N/A	24	0.0				SP	Same as above	
82	N/A	24	0.0				GW	GRAVEL Brown, fine, medium-coarse sand (30%), wet	
84	N/A	24	0.0				SW	SAND Brown, medium-coarse, fine gravel (30%), well graded, wet	
86	N/A	24	0.0				SW	Same as above	
86	N/A	24	0.0				SP	SAND Olive gray, fine-medium, poorly graded, wet	
86	N/A	24	0.0				SP	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 7 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
88	N/A	24	0.0				SP	Same as above	
90	N/A	24	0.0				SP	Same as above	
92	N/A	24	0.0				SP	Same as above	
94	N/A	24	0.0				SP	Same as above	
96	N/A	24	0.0				SP	Same as above	
98	N/A	24	0.0				SP	Same as above, gray	
100	N/A	24	0.0				SP	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 8 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648  
 Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630  
 Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58  
 Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
102	N/A	24	0.0				SW	SAND Brown, fine-coarse, trace fine-medium gravel, well graded, wet	
104	N/A	24	0.0				SW	Same as above	
106	N/A	24	13.1				GW	GRAVEL Gray, fine-medium, fine-coarse sand (30%), well graded, wet	
108	N/A	24	13.2				GW	Same as above	
110	N/A	24	2.4				GW	Same as above	
112	N/A	24	0.7				GW	Same as above	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 9 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coor.: 3450.80045     East Coor.: 7183.08786

Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
114	N/A	24	2.1				GW	Same as above	
116								End of boring	
118									
120									
122									
124									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 10 of 10

Drilling Co.: Boart Longyear     Geologist: T. Fortner     Begin Drilling: 8/21/06 @ 1648

Driller: K. Gobell     Total Depth: 115     End Drilling: 8/22/06 @ 1630

Drilling Method: Rotosonic     Surface Elev.: 735.588     Converted to Well: Y Well I.D.: GM-58

Drilling Fluid: Water     North Coord.: 3450.80045     East Coord.: 7183.08786

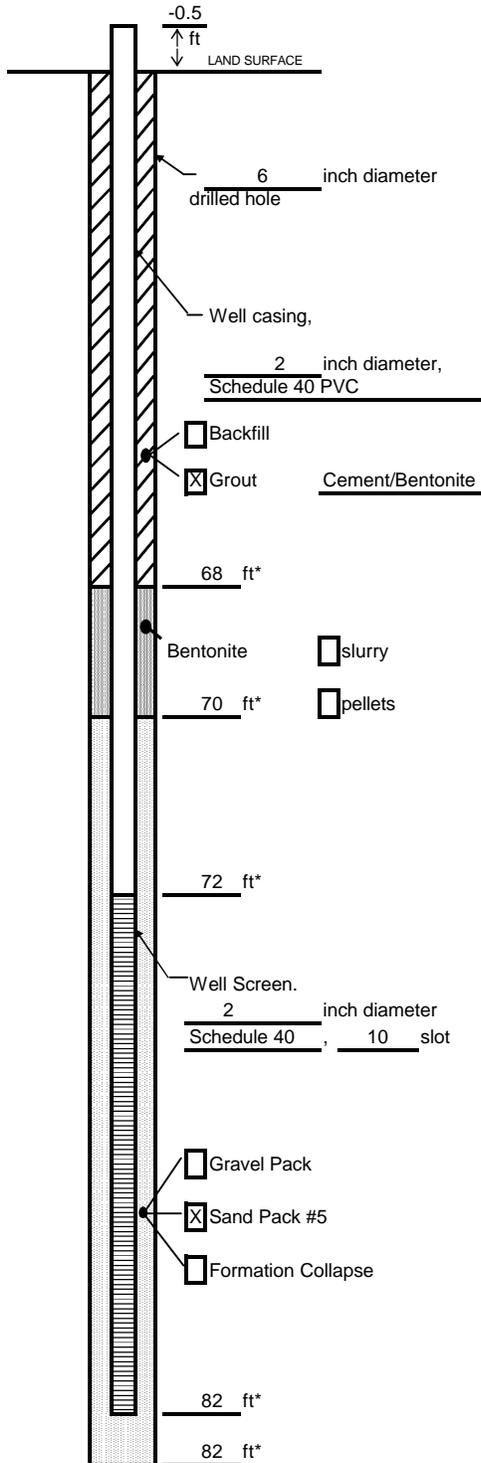
Remarks: Water samples 25-30'; 75-80'; 95-100'

Project No.: OH000294.0009     Datum: TOC Elev=735.462     Filename: August 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.  
\* Depth Below Land Surface

Project General Motors Corporation Well GM-58

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

735.588 feet  Surveyed

Estimated

Installation Date(s) 9/1/2006

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)

Pumping - surge with pump 9/1/06

Fluid Loss During Drilling NM gallons

Water Removed During Development NM gallons

Static Depth to Water 28.32 feet below M.P.

Pumping Depth to Water 79 feet below M.P.

Pumping Duration NM hours

Yield NM gpm Date NA

Specific Capacity NM gpm/ft

Well Purpose Monitoring Well - Well I

Remarks TOC Elevation = 735.462

Time 1600, 1610, 1615, 1620, 1625, 1630

pH 6.78, 6.61, 6.61, 6.58, 6.59, 6.59

Conductivity 1.20, 1.20, 1.20, 1.21, 1.20, 1.20

Turbidity 232, 137, 72, 36, 28, 18

Temp 17.8, 17.3, 17.5, 17.4, 17.3, 17.4

Prepared by J. Manzo/ J. Wallace

General Motors Corporation	Moraine, Ohio
----------------------------	---------------

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0								See RZ-4J for lithologic description from 0-40'	
2									
4									
6									
8									
10									
12									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 1

Drilling Co.: Boart Longyear    
 Geologist: J. Wallace    
 Begin Drilling: 8/31/06

Driller: D. Remmler    
 Total Depth: 40    
 End Drilling: 8/31/06

Drilling Method: Hollow Stem Auger    
 Surface Elev.: 726.207    
 Converted to Well: Y Well I.D.: GM-63

Drilling Fluid: Water    
 North Coord.: 1625.02787    
 East Coord.: 4918.82945

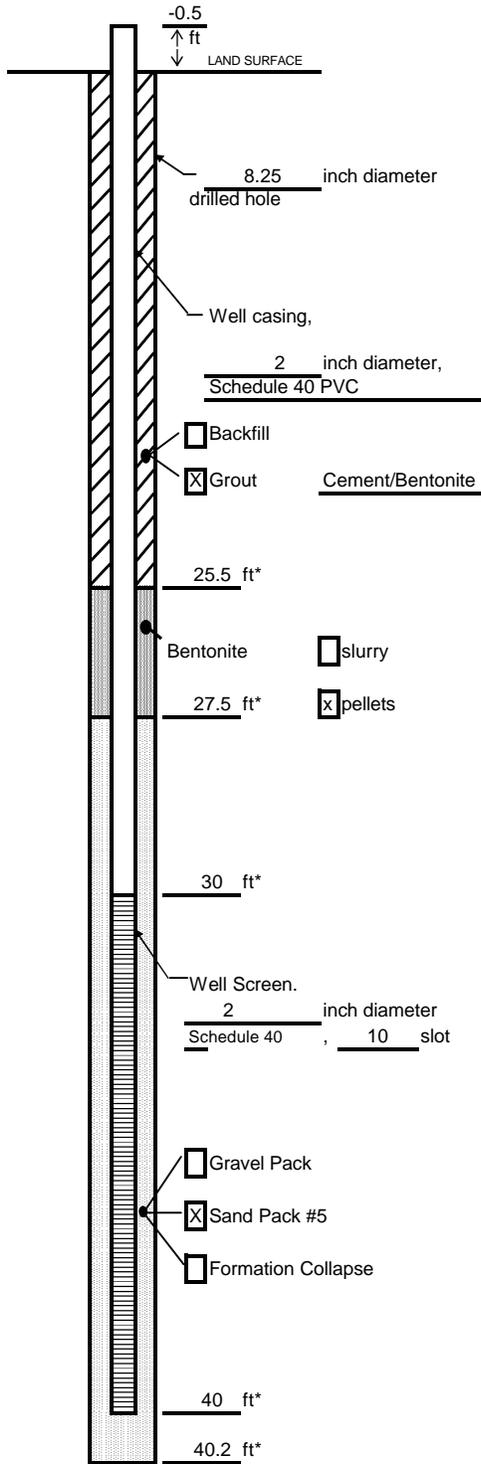
Remarks: Shallow pair to GM-64.

Project No.: OH000294.0008.00002    
 Datum: TOC Elev. 725.791    
 Filename: July 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.  
\* Depth Below Land Surface

Project General Motors Corporation Well GM-63

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

726.207 feet  Surveyed

Estimated

Installation Date(s) 9/1/2006

Drilling Method Hollow Stem Auger

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)

Pumping - surge with pump 9/1/06

Fluid Loss During Drilling 15 gallons

Water Removed During Development 50 gallons

Static Depth to Water ~20.2 feet below M.P.

Pumping Depth to Water 38 feet below M.P.

Pumping Duration NA hours

Yield ~1.6 gpm Date 9/1/06

Specific Capacity NM gpm/ft

Well Purpose Monitoring Well

WSU-22 Replacement (Shallow)

Remarks TOC Elevation = 725.791

Time 13:40, 13:45, 13:50, 13:55, 14:00

pH 6.65, 6.58, 6.58, 6.58, 6.59

Conductivity 1.41, 1.41, 1.42, 1.41, 1.41

Turbidity 588, 286, 157, 92, 59

Temperature 18.8, 18.6, 18.6, 18.5, 18.5

Prepared by J. Wallace

General Motors Corporation	Moraine, Ohio
----------------------------	---------------

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0								See RZ-4J for lithologic description from 0-60'	
2									
4									
6									
8									
10									
12									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 1

Drilling Co.: Boart Longyear    
 Geologist: J. Wallace    
 Begin Drilling: 8/31/06

Driller: D. Remmler    
 Total Depth: 60    
 End Drilling: 8/31/06

Drilling Method: Hollow Stem Auger    
 Surface Elev.: 726.384    
 Converted to Well: Y Well I.D.: GM-64

Drilling Fluid: Water    
 North Coord.: 1624.73847    
 East Coord.: 4913.72063

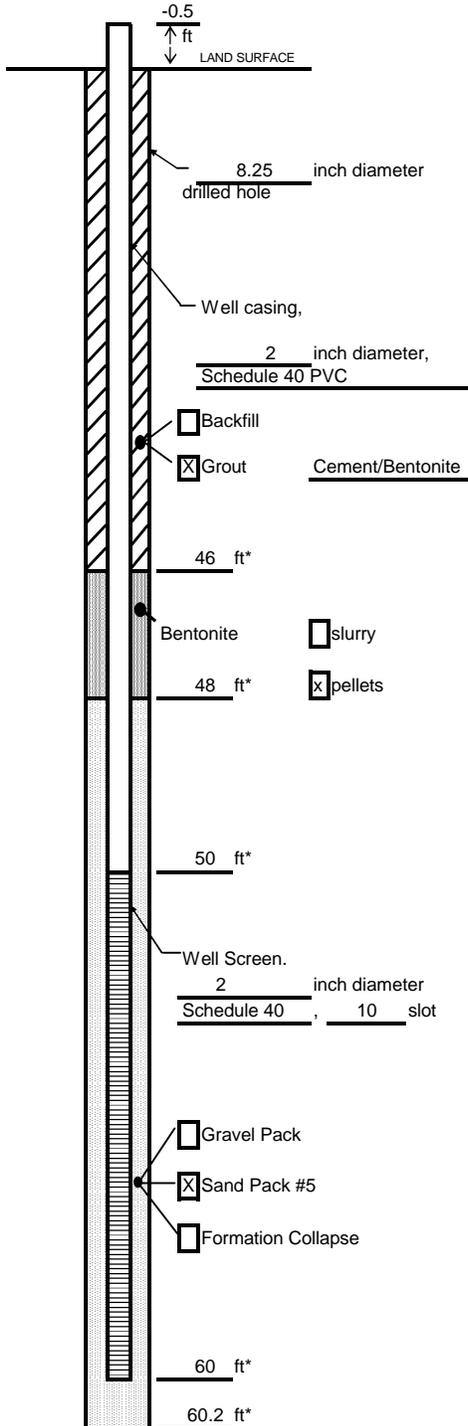
Remarks: Shallow pair to GM-54.

Project No.: OH000294.0008.00002    
 Datum: TOC Elev. 725.951    
 Filename: July 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.  
\* Depth Below Land Surface

Project General Motors Corporation Well GM-64

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

726.384 feet  Surveyed

Estimated

Installation Date(s) 8/31/2006

Drilling Method Hollow Stem Auger

Drilling Contractor Boart Longyear

Drilling Fluid None. However, ~25 gallons water

added due to heaving sands

Development Technique(s) and Date(s)

Pumping - surge with pump

Fluid Loss During Drilling 25 gallons

Water Removed During Development 55 gallons

Static Depth to Water 20.4 feet below M.P.

Pumping Depth to Water 58 feet below M.P.

Pumping Duration 0.85 hours

Yield NM gpm Date 8/31/06

Specific Capacity NM gpm/ft

Well Purpose Monitoring Well

WSU-22 Replacement (Deep)

Remarks TOC Elevation = 725.951

Time 13:00, 13:05, 13:10, 13:15, 13:20, 13:25

pH 6.78, 6.64, 6.64, 6.67, 6.66, 6.67

Conductivity 1.29, 1.28, 1.28, 1.29, 1.29, 1.29

Turbidity 10+, 10+, 999, 999, 999, 10+

Temperature 19.7, 19.7, 19.6, 19.5, 19.6, 19.6

Pumped 25 gallons prior to taking readings

Prepared by J. Wallace

General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0								No recovery	
2								No recovery	
4	N/A	24	0.0				SW	SAND Brown, fine-medium, medium-coarse gravel (15%), loose, dry	
6	N/A	24	0.7				SW	SAND Light grayish brown, fine-medium, medium-coarse gravel (15%), loose, dry	
8	N/A	24	0.3				SW	Same as above	
10	N/A	24	2.7				SW	Same as above	
12									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 1 of 6

Drilling Co.: Boart Longyear

Geologist: C. Cattlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coord.: 1690.15438

East Coord.: 4887.95475

Remarks: \_\_\_\_\_

Project No.: OH000294.0009.0004B

Datum: TOC Elev. 727.965

Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
12	N/A	24	2.4				SW	Same as above	
14	N/A	24	5.3				SW	Same as above	
16	N/A	24	5.1				SW	Same as above	
18	N/A	24	5.2				SW	Same as above	
20	N/A	24	2.7				SW	Same as above	
22	N/A	24	1.6				SW	Same as above	
24									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 2 of 6

Drilling Co.: Boart Longyear

Geologist: C. Catlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coor.: 1690.15438

East Coor.: 4887.95475

Remarks: \_\_\_\_\_

Project No.: OH000294.0009.0004B

Datum: TOC Elev. 727.965

Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
24	N/A	24	4.7				SW	Same as above	
26	N/A	24	3.9				SW	SAND Brown, coarse sand with fine-medium gravel (20%), loose, wet	11
28	N/A	24	3.3				SW	Same as above	
30	N/A	24	8.7				SW	Same as above	
32	N/A	24	9.4				SW	Same as above	
34	N/A	24	6.6				SP	SAND Brown, medium sand, loose, wet	
36									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 3 of 6

Drilling Co.: Boart Longyear

Geologist: C. Catlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coord.: 1690.15438

East Coord.: 4887.95475

Remarks: \_\_\_\_\_

Project No.: OH000294.0009.0004B

Datum: TOC Elev. 727.965

Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
36	N/A	24	0.0				SP	Same as above	
38	N/A	24	5.6				SW	SAND Brown, sand and gravel (20%), loose, wet	
40	N/A	24	9.6				SP	SAND Brown, medium sand, loose, wet	
42	N/A	24	0.0				SP	Same as above	
44	N/A	24	6.2				SP	Same as above	
46	N/A	24	0.0				SW	Same as above, gravel (10%)	
48									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 4 of 6

Drilling Co.: Boart Longyear

Geologist: C. Catlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coord.: 1690.15438

East Coord.: 4887.95475

Remarks: \_\_\_\_\_

Project No.: OH000294.0009.0004B

Datum: TOC Elev. 727.965

Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
48	N/A	24	5.8				SW	SAND Brown, medium-coarse sand, loose, wet	
50	N/A	24	0.0				SW	SAND Brown, sand with gravel (5-10%), loose, wet	
52	N/A	24	5.8				SW	Same as above	
54	N/A	24	0.0				SW	Same as above	
56							ROCK	Boulder	
58							ROCK	Same as above	
60							ROCK	Same as above	

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 5 of 6

Drilling Co.: Boart Longyear

Geologist: C. Cattlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coord.: 1690.15438

East Coord.: 4887.95475

Remarks:

Project No.: OH000294.0009.0004B

Datum: TOC Elev. 727.965

Filename: July 2006

### General Motors Corporation

Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
60	N/A	12	0.0				ROCK	Same as above	
62	N/A	24	4.1				CL	SILTY CLAY Gray, little sand, trace gravel, dense	
64	N/A	12	4.0				CL	Same as above	
66								End of boring	
68									
70									
72									

Composite Sample to Lab

Grab Sample to Lab

Split-Spoon Not Analyzed

Page 6 of 6

Drilling Co.: Boart Longyear

Geologist: C. Catlett

Begin Drilling: 7/16/06 @ 1635

Driller: M. Osterberg

Total Depth: 65

End Drilling: 7/16/06 @ 1810

Drilling Method: Rotosonic

Surface Elev.: 724.428

Converted to Well: Y Well I.D.: RZ-4J

Drilling Fluid: Water

North Coord.: 1690.15438

East Coord.: 4887.95475

Remarks: \_\_\_\_\_

Project No.: OH000294.0009.0004B

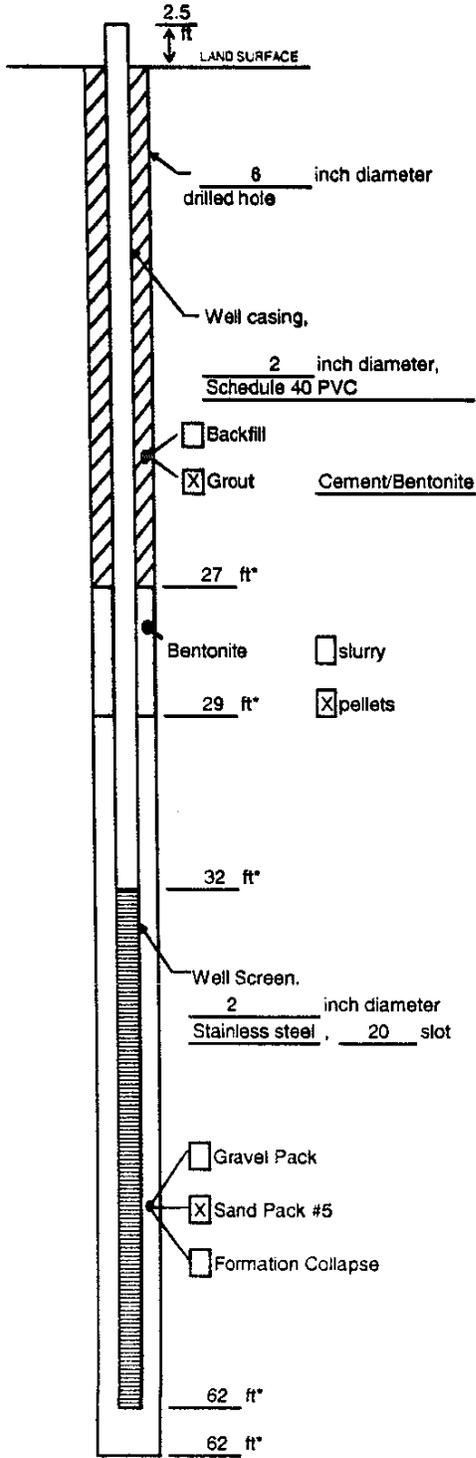
Datum: TOC Elev. 727.965

Filename: July 2006

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.

\* Depth Below Land Surface

Project General Motors Corporation Well RZ-4J

Town/City Moraine

County Montgomery State Ohio

Permit No. NA

Land-Surface (LS) Elevation and Datum:

724.262 feet  Surveyed

Estimated

Installation Date(s) 7/16-7/17/06

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)

Pumping - surge with pump 7/29/06

Fluid Loss During Drilling -200 gallons

Water Removed During Development 240 gallons

Static Depth to Water 21.66 feet below M.P.

Pumping Depth to Water NM feet below M.P.

Pumping Duration 1.00 hours

Yield 4 gpm Date 7/29/06

Specific Capacity NM gpm/ft

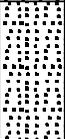
Well Purpose Introduction well

Remarks TOC Elevation = 727.965

Clear non-turbid

Prepared by J. Manzo

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
0	N/A	N/A	N/A				FILL	FILL Utility cleared material	
2									
4									
6									
8									
10									
12	N/A	24	7.8				SP	SAND Yellowish brown (10 yr 5/6), poorly graded, trace silt, fine-medium grained, 20% gravel (angular-subround), dry	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 1 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
14	N/A	24	7.0						
16	N/A	24	7.9					Note: Coarse sand, yellowish brown (10 yr 5/6), 30% gravel @ 15'	
18	N/A	24	7.2						
20	N/A	24	9.2						
22	N/A	24	6.2						
24	N/A	24	11.4				SP	SAND Yellowish brown (10 yr 5/4), poorly graded, <5% gravel (subangular-	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 2 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
26	N/A	24	10.2			[Dotted pattern]		subround), fine medium, dry Note: Yellowish brown (10 yr 5/6), 30% gravel, coarse, moist	
28	N/A	24	8.7			[Dotted pattern]	GP	GRAVEL WITH SAND Yellowish brown (10 yr 5/6), poorly graded, medium grained, 20% sand, subangular-angular, wet	
30	N/A	24	8.0			[Dotted pattern]			
32	N/A	24	6.9			[Dotted pattern]			
34	N/A	24	7.6			[Dotted pattern]			
36	N/A	24	8.0			[Dotted pattern]	SP	SAND Yellowish brown (10 yr 5/4), poorly graded, medium coarse, 10% gravel (subangular-subround), wet	
	N/A	24	6.1			[Dotted pattern]	GW	GRAVEL WITH SAND Brown (10 yr 5/4), well graded, fine medium 10% sand, subangular-	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 3 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water	
38	N/A	24	7.5					subround, wet		
40	N/A	24	7.2					Water sample 40-45' @ 1150		
42	N/A	24	6.8					Note: Fine-coarse, sandy gravel, 10% sand		
44	N/A	24	7.4							
46	N/A	24	1.5					CL	SILTY CLAY Dark grayish brown (10 yr 4/2), 5% gravel (angular-subangular), very stiff, low plasticity, dry	
48	N/A	24	4.7							
50										

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 4 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
52	N/A	24	3.9				ML	CLAYEY SILT Dark grayish brown (10 yr 4/2), <5% gravel (subround), moist	
54	N/A	24	4.3				CL	SILTY CLAY Dark grayish brown (10 yr 4/2), 5% gravel (subangular-angular), very stiff, brittle, low plasticity, dry	
56	N/A	24	4.8						
58	N/A	24	5.0						
60	N/A	24	4.6						
62	N/A	24	5.1						

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 5 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
64	N/A	24	5.6						
66	N/A	24	4.7				SP	SAND Dark grayish brown (10 yr 4/2), poorly graded, fine-medium, <5% fine gravel, moist	
68	N/A	24	3.4				CL	SILTY CLAY Dark grayish brown (10 yr 4/2), 5% gravel, subangular-angular, very stiff, brittle, low plasticity, dry	
70	N/A	24	5.1						
72	N/A	24	4.8						
74	N/A	24	4.4				SP	SAND Yellowish brown (10 yr 5/4), poorly graded, 15% gravel (subangular-angular), medium-coarse, wet	
	N/A	24	5.2					Water sample 73-78' @ 1520	

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 6 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
76	N/A	24	5.2						
78	N/A	24	7.0					Note: 25% gravel (subangular-subround) @ 79'	
80	N/A	24	6.1				ML	CLAYEY SILT Yellowish brown (10 yr 5/4), orange mottling, dry	
82	N/A	24	7.8				SP	SAND Dark gray (10 yr 4/1), poorly graded, fine, wet	
84	N/A	24	4.5					Note: Dark grayish brown (10 yr 4/2), <5% gravel @ 83'	
86	N/A	24	5.6						

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 7 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
88	N/A	24	7.5					Note: 15-20% gravel @ 88'	
90	N/A	24	7.6					Note: 5% Gravel, trace silt	
92	N/A	24	6.7				SW	SAND Dark grayish brown (10 yr 4/2), well graded, fine-coarse, 5% gravel, moist, trace silt, seams of fine sand <2", wet  Water sample 93-98' @ 1650	
94	N/A	24	3.6						
96	N/A	24	7.0						
98	N/A	24	5.7					Note: 15% gravel, no fine sand seams @ 98'	
100									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 8 of 10

Drilling Co.: Boart Longyear     Geologist: L. Greene     Begin Drilling: 4/5/2007  
 Driller: Gerald Sealey     Total Depth: 120     End Drilling: 4/5/2007  
 Drilling Method: Rotosonic     Surface Elev.: 737.470     Converted to Well: Y Well I.D.: GM-70  
 Drilling Fluid: Water     North Coord.: 3887.32724     East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010     Datum: TOC: 737.186     Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (/6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
102	N/A	24	7.1					Note: 25-30% fine gravel, fine-coarse, trace silt @ 102'	
104	N/A	24	6.8						
106	N/A	24	5.4						
108	N/A	24	5.1						
110	N/A	24	4.2				SP	SAND Dark grayish brown (10 yr 4/2), poorly graded, fine-coarse, 5% gravel (subangular-subround), wet	
112	N/A	24	2.1						Water sample 112-117' @ 1810

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 9 of 10

Drilling Co.: Boart Longyear    
 Geologist: L. Greene    
 Begin Drilling: 4/5/2007

Driller: Gerald Sealey    
 Total Depth: 120    
 End Drilling: 4/5/2007

Drilling Method: Rotosonic    
 Surface Elev.: 737.470    
 Converted to Well: Y Well I.D.: GM-70

Drilling Fluid: Water    
 North Coord.: 3887.32724    
 East Coord.: 7282.84039

Remarks: \_\_\_\_\_

Project No.: OH000294.0010    
 Datum: TOC: 737.186    
 Filename: April 2007

**General Motors Corporation** Moraine, Ohio

Depth (feet)	Blows (6 in.)	Recovery (Inches)	OVA (PPM)	Sample Analysis	Sample Type	Graphic Log	Soil Class.	Description	Depth to Water
114	N/A	24	3.7						
116	N/A	24	3.5						
118	N/A	24	5.5				CL	SILTY CLAY Dark gray (10 yr 4/1), trace fine gravel (fossiliferous limestone), stiff, dry	
120	N/A	12	2.2						
122								End of boring	
124									

Composite Sample to Lab    
  Grab Sample to Lab    
  Split-Spoon Not Analyzed    
 Page 10 of 10

Drilling Co.: Boart Longyear    
 Geologist: L. Greene    
 Begin Drilling: 4/5/2007

Driller: Gerald Sealey    
 Total Depth: 120    
 End Drilling: 4/5/2007

Drilling Method: Rotosonic    
 Surface Elev.: 737.470    
 Converted to Well: Y Well I.D.: GM-70

Drilling Fluid: Water    
 North Coord.: 3887.32724    
 East Coord.: 7282.84039

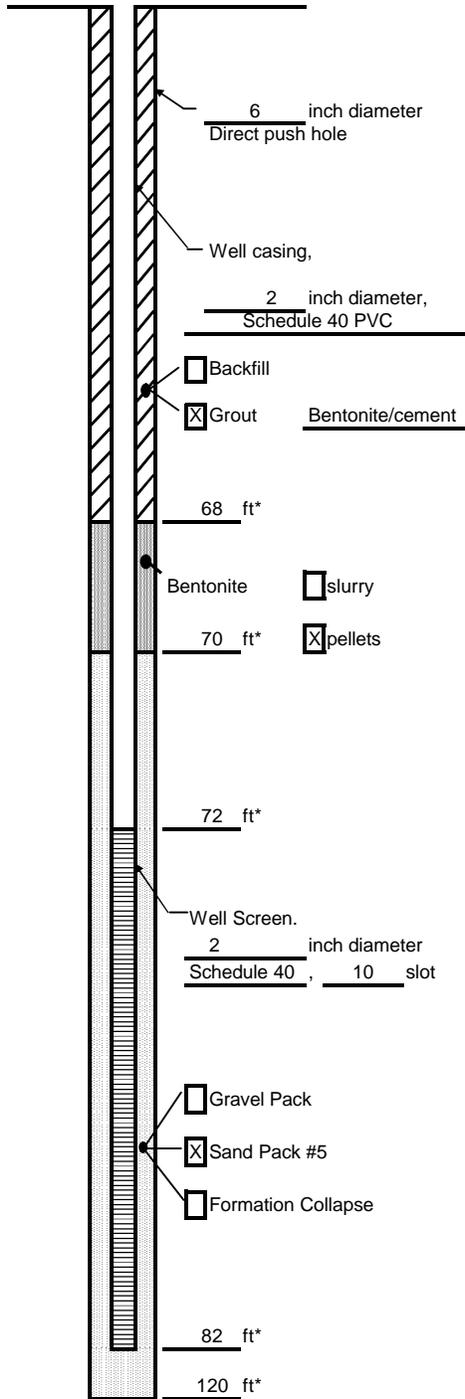
Remarks: \_\_\_\_\_

Project No.: OH000294.0010    
 Datum: TOC: 737.186    
 Filename: April 2007

**ARCADIS**

**Well Construction Log**

(Unconsolidated)



Measuring Point is  
Top of Well Casing  
Unless Otherwise Noted.

\* Depth Below Land Surface

Project General Motors Corporation Well GM-70

Town/City Moraine

County Montgomery State Ohio

Permit No. N/A

Land-Surface (LS) Elevation and Datum:  
737.470 feet  Surveyed  
 Estimated

Installation Date(s) 4/12/2007

Drilling Method Rotosonic

Drilling Contractor Boart Longyear

Drilling Fluid Water

Development Technique(s) and Date(s)

Submersible pump, surge with pump

4/13/07 Paul Smith

Fluid Loss During installation 50 gallons

Water Removed During Development 60 gallons

Static Depth to Water 26.5 feet below M.P.

Pumping Depth to Water NM feet below M.P.

Pumping Duration 0.50 hours

Yield 3 gpm Date 4/13/07

Specific Capacity NM gpm/ft

Well Purpose GW Monitoring

Note: formation collapse 120-82'

Remarks Time: 1219, 1222, 1225, 1228, 1231

pH: 7.49, 7.84, 7.94, 7.99, 8.01

Conductivity: 1.09, 1.08, 1.06, 1.05, 1.06

Turbidity: 17, 4, 10+, 10+, 10+

Temperature: 15.7, 15.7, 15.8, 15.9, 15.8

Prepared by L. Greene