

**ATTACHMENT E-2**  
**SAMPLING AND ANALYSIS PLAN (SAP)**

**SAMPLING AND ANALYSIS PLAN  
VEOLIA CLOSURE PROJECT**

**Phoenix, Arizona**

*Prepared for  
Veolia Environmental Solutions*

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## 1.0 INTRODUCTION

This Sampling and Analysis Plan (SAP) presents the requirements and procedures for performing sampling activities associated with the closure of the former polychlorinated biphenyl (PCB) storage and processing facility located at 5736 West Jefferson (Figure 1) and operated by Veolia Environmental Services (VES or Veolia). This project-specific SAP has been prepared to ensure that:

- Appropriate quality assurance measures for the project are defined and met;
- Field sampling associated with the project follows consistent protocols and field activities are fully documented; and
- The data collected meet project objectives and are both scientifically valid and defensible.

This SAP is a companion document to the Closure Plan prepared to address the Toxic Substances Control Act (TSCA) requirements for closing a PCB storage facility identified in Title 40, Code of Federal Regulations (CFR) Part 761.65(e). The Closure Plan is presented in VES' TSCA Application dated October 2012.

This SAP presents detailed information supporting data collection, analysis, and interpretation for the purpose of demonstrating clean closure of the interiors of Buildings 2, 3, and 4, the walls of the loading docks, the ground surfaces surrounding the three buildings, six sealed dry wells, and two open dry wells located in the vicinity of the three buildings that may have been impacted by PCB-related operations carried out at the facility.

### 1.1 BACKGROUND

As documented in the TSCA Application, VES is a site that has historically managed PCBs and PCB materials. VES wishes to provide a closure plan and this SAP to document how data will be collected, reviewed, and presented in support of site closure activities when they occur. The project area includes the interiors of the three buildings discussed above, the walls of the loading docks, the ground surfaces in proximity to the buildings and outside storage areas, and the eight aforementioned dry wells. Buildings 2 and 3 were used for storage of PCB materials, as well as processing facilities. PCB storage activity was historically conducted in only a portion of Building 4 with no processing of PCBs being performed in the building interior.

Additional site description and history information are presented in the TSCA Application.

## 1.2 PROJECT OBJECTIVES

The overall objectives of the facility closure project are:

- To assess and address compliance with 40 CFR 761.65(e) for PCB storage closure activities by determining if surfaces meet the clean closure requirements of 40 CFR 761 Subpart G.

The constituents of potential concern (COPC) for the project are PCBs, based on site history and known activities.

## **2.0 QUALITY ASSURANCE**

Project quality assurance (QA) will be maintained by integrating industry standard best practices into the data collection, analysis, and assessment activities conducted to verify clean closure of the site in accordance with the standards set forth under TSCA. An overview of key project QA elements is presented in this section.

### **2.1 DATA QUALITY OBJECTIVE PROCESS**

To ensure that the appropriate type, quality, and quantity of data are collected to support achieving project objectives, the Data Quality Objective (DQO) process for environmental site investigation activities will be implemented. This seven step process identifies the nature of the problem that collected data are intended to address, required decisions that need to be made, inputs into the decision (including constraints, schedule requirements, project boundaries, necessary data), how the data will be used to make the decision, and optimization of a sampling design developed to collect the required data.

For the VES Project, development of the sampling program was subject to the DQO process. The DQOs are presented in Table 1. Implementation of this process for clean-up verification activities is presented in Section 3.0 of this SAP.

### **2.2 STANDARD OPERATING PROCEDURES FOR FIELD ACTIVITIES**

Consistency in data collection will be maintained throughout field activities via strict adherence to project Standard Operating Procedures (SOPs) by an experienced team of field personnel. Appendix A presents the SOP for collection of samples from porous surfaces, Appendix B presents the SOP for collection of wipe samples from non-porous surfaces, and Appendix C includes additional project SOPs for reference during implementation of this SAP:

- Field Activity Records
- Chain-of-Custody Forms
- Sample Management
- Collection of Soil Samples
- Investigation-Derived Waste

## **2.3 ANALYTICAL QUALITY ASSURANCE PROGRAM**

Collected environmental samples will be analyzed by laboratories that have well-developed QA programs; the laboratories used to implement this SAP will be licensed by the Arizona Department of Health Services (ADHS) to perform the analyses specified herein (as applicable). ADHS routinely reviews compliance of licensed laboratories through annual on-site audits and proficiency evaluation sample analysis.

Laboratory Quality Control (QC) measures for standards, blanks, calibration, analyte identification and quantitation will be performed as specified by the analytical standard method, the laboratory Quality Assurance Plan, and this SAP (as applicable).

## **2.4 USE OF DATA QUALITY INDICATORS IN DATA ASSESSMENT**

The data quality indicators (DQIs) of precision, accuracy, completeness, comparability and representativeness are routinely used to assess the QC of environmental sample collection and analytical procedures. For the VES Project, the field sampling program will include the collection of field QC samples at appropriate frequencies to support this assessment (see Section 3.2.4 for QC sampling frequencies). Additional information regarding DQIs with associated QA acceptance criteria for the project is presented in Appendix D.

## **3.0 SAMPLING PROGRAM DESCRIPTION**

### **3.1 SAMPLING PLAN DEVELOPMENT**

The Site Characterization field sampling plan was developed using the DQO Process to identify the appropriate type, quality, and quantity of data to be collected in support of project objectives. Table 1 summarizes DQO process implementation.

### **3.2 SAMPLING PROGRAM ELEMENTS AND RATIONALE**

#### **3.2.1 Sample Analyte, Sampling Media, and Applicable Regulatory Standards**

PCBs are the analytes of concern. In this case, building interior and exterior surfaces, ground surfaces, and sealed dry wells are considered to be the media to be sampled. Building interior surfaces consist of porous surfaces (i.e., concrete floor and block wall) and non-porous surfaces (i.e., doors and ventilation fans). Building exterior surfaces include porous surfaces (i.e., block wall) and non-porous surfaces (i.e., doors). Ground surfaces include porous surfaces (i.e., concrete and asphalt). Sampling of the sealed dry wells will initially consist of sediments in the bottom of the dry well, and depending on the analytical results of the sediment sampling, soil samples collected from borings in the vicinity of any of the sealed dry wells for which sediment results exceed the closure criteria.

The historic activities conducted within Building 4 (storage only) are not expected to have generated dust. Therefore, air sampling is not anticipated to be necessary within this building. Prior to conducting any closure activities within Buildings 2 and 3, dust samples will be collected from the upper confines of those areas where PCB processing activities took place that may have created hazardous fugitive dust (i.e., the southeast corner of Building 2 and the southwest corner of Building 3). If the analyses of these dust samples demonstrate that a hazard exists, appropriate mitigations will be implemented in accordance with the project health and safety plan as directed by the health and safety officer.

In accordance with 40 CFR 761.65(e), the applicable regulatory standard is 1 part per million (ppm) for porous materials, soil, and sediment and 10 micrograms per 100 square centimeters for non-porous materials. These standards are based upon the PCB Spill Clean-up policy codified at 40 CFR 761, Subpart G, as referenced by 40 CFR 761.65(e). Please note that the standards for non-porous surfaces are clearly stated throughout the TSCA regulations; however, those for porous surfaces are not directly stated in Subpart G, but were implied from the soil cleanup

levels stated in that part [see 40 CFR 761.125(c)(4)(v)]. Conformance with the clean-up standards of Subpart G [40 CFR 761.125(c)(4)(i) through (v)] are verified through the implementation of post-cleanup sampling specified in 40 CFR 761.130. This SAP was developed to assure compliance with that part.

### **3.2.2 Sampling Program Extent**

#### **3.2.2.1 Building 2**

Sampling will be conducted at locations where PCB activities, including receiving and storage, are expected to occur within Building 2. This includes storage locations, the 10-day transfer area, and the PCB receiving area within the building. No processing of PCBs occurred in this building. To that end, Figure 2 identifies where PCB activities were known to occur, and areas that may have been impacted by movement of PCBs within the building. The sampling program extent includes the potentially impacted floor footprint and wall areas adjacent to the floor footprint where PCB handling activities occurred to a height of up to 10 feet. The thresholds of any doors falling outside of the floor footprint and blade surfaces of the four ventilation fans in Building 2 will also be included within the sampling program extent. In addition, a dust sample will be collected from elevated surfaces in the southeast corner of Building 2 prior to closure activities to assess whether mitigation measures are required to preclude exposure of personnel to hazardous fugitive dust.

#### **3.2.2.2 Building 3**

Sampling will be conducted at locations where PCB activities, including storage and processing, occurred within Building 3. This includes the PCB storage areas and the processing area. Figure 3 identifies where PCB activities were known to occur, and includes areas that may have been impacted by movement of PCBs within the building. The sampling program extent includes the potentially impacted floor footprint and wall areas adjacent to the floor footprint to a height of up to 10 feet. The thresholds of any doors falling outside of the floor footprint and blade surfaces of the three ventilation fans in Building 3 will also be included with the sampling program extent. In addition, a dust sample will be collected from elevated surfaces in the southwest corner of Building 3 prior to closure activities to assess whether mitigation measures are required to preclude exposure of personnel to hazardous fugitive dust.

#### **3.2.2.3 Building 4**

Sampling will be conducted at locations where PCB activities could reasonably have been expected to occur within Building 4. This includes storage locations and traffic routes

(movement of PCBs and PCB items) within the building. No processing of PCBs occurred in this building. To that end, Figure 4 identifies where PCB activities were known to occur, and areas that may have been impacted by movement of PCBs within the building. The sampling program extent includes a floor footprint of 116.5 feet by 30 feet (incorporating the eastern side of the building where PCBs were stored or moved) and wall areas adjacent to the floor footprint where PCB handling activities occurred to a height of up to 10 feet. The thresholds of any doors falling outside of the proposed floor footprint will also be included within the sampling program extent.

#### ***3.2.2.4 Loading Dock Walls***

Sampling of the loading dock wall surfaces will be conducted because of the potential to have been impacted by PCB materials delivery operations. Figure 5 provides a sampling schematic for the loading dock walls. Sampling of the floor surfaces of the loading docks will be covered under the ground surface sampling requirements. For Building 4, a minimum of three samples will be collected from the building's exterior wall surrounding the portal through which PCBs may have been pumped. This portal is located approximately 6 feet from the south entrance into the loading dock at a height of 6 feet above ground surface.

#### ***3.2.2.5 Ground Surfaces***

Sampling will be conducted at ground surface locations where PCB activities could reasonably have been expected to have impacted the ground surfaces in the vicinity of Buildings 2, 3, and 4. This includes the loading docks and the ground surfaces immediately adjacent to the loading docks, as well as the entire ground surfaces located between the loading docks adjacent to Buildings 2 and 3, the ground surfaces between Buildings 2, 3, and 4 extending to the northern boundary of the facility, and those ground surfaces extending south of the facility fence incorporating areas surrounding the two open dry wells as shown in Figure 6.

#### ***3.2.2.6 Dry Wells***

Three discrete sediment samples will be collected from the settling chamber within each of the six sealed and two open dry wells delineated in Figure 6. If it is determined for an individual dry well that the total PCB concentration for the sediment samples exceeds 1 ppm, a boring will be advanced at a distance of five feet from the dry well and soil samples collected every five feet and at any distinct lithology changes starting at the depth of the bottom of the settling chamber and continuing to a total depth of at least 10 feet below the bottom of the drywell injection pipe.

### 3.2.3 Number of Samples and Planned Sampling Locations

Table 1 summarizes the sampling design that will be used to evaluate whether clean closure has occurred and Figures 2, 3, 4, 5, and 6 present planned sampling locations for Building 2, Building 3, Building 4, the loading dock walls, and the ground surfaces, respectively. Figure 6 also provides the locations of the eight dry wells that will be sampled. The number of samples to be collected during closure were determined using Visual Sample Plan (VSP) Version 6.0 and based upon the requirements of 40 CFR 761.130. Discrete chip, wipe, sediment and soil samples will be collected; composite sampling is not proposed at this time. Samples will be collected from each area using a sampling grid based on the areal extent of the site PCB activities. The number of samples collected is derived from the minimum sample number recommended by VSP plus quality control samples.

Planned sampling locations of building interiors are based on the sampling grid established using VSP and include the building floors footprint and wall areas adjacent to the floor footprint to a height of up to 10 feet. The doors are included in the wall area for purposes of the sampling grid placement; however, the type of sample that will be collected will vary based on whether the sampling point is placed on a door or wall. In addition, judgmental samples will be placed within the threshold (floor) of doorways as shown in the sampling figures. For Buildings 2 and 3, dust samples will be collected prior to implementation of closure activities in those buildings as discussed previously. In addition, wipe samples will be collected from the blade surfaces of the four ventilation fans located in Building 2 and the three ventilation fans located in Building 3.

The walls of the loading docks located at Buildings 2, 3, and 4 will be sampled, as well as wall surfaces surrounding the portal at Building 4 as described above. Each loading dock is 60 ft long and varies in depth from ground surface to 4 ft below ground surface.

The ground surfaces will include the loading docks, as well as the ground surface located between Buildings 2 and 3, the ground surface located between Building 4 and 25 ft to the east of the building, the ground surfaces extending north from Buildings 2, 3, and 4 to the facility boundary, and the ground surfaces extending south from the delivery areas incorporating the footprints of the two open dry wells located there. The sample type will vary depending on the surface to be sampled (e.g., concrete, asphalt).

The three sealed dry wells located within the footprints of the loading docks at Buildings 2, 3, and 4, along with the three sealed dry wells located within 30 feet of the north corners of the three buildings will be sampled. Also, the two open dry wells located just south of the delivery areas between Buildings 2 and 3 and east of Building 4 will be sampled. The three sealed dry

wells located with the loading docks are underneath concrete and will require removal of the concrete to expose the dry wells. The three sealed dry wells located just to the north of the buildings have been sealed by welded metal plates that will be removed prior to sampling. Initially, the sediment at the bottom of the settling chambers will be sampled with additional borehole sampling required should the PCB sediment concentration exceed 1 ppm for a given dry well.

The total numbers and types of samples to be collected are provided in Table 2.

### **3.2.4 Frequency of Quality Control Sampling**

QC samples to be collected during closure include:

- Field Duplicate (FD) samples will be collected at a rate of 10% (170 field duplicate samples are anticipated during closure).
- One Matrix Spike/Matrix Spike Duplicate (MS/MSD) sample per 20 samples for PCB analysis (78 MS samples are anticipated during closure).
- One laboratory Duplicate (D) sample per 20 samples will be analyzed for PCBs (78 D samples are anticipated during closure).
- Equipment blanks (EB) will be collected at a frequency of one per sampling day.

All sample coolers are anticipated to contain temperature blanks (i.e., glass or poly bottles containing water), which accompany the samples shipped to the laboratory, to be used by the laboratory to measure the temperature of the cooler upon receipt.

### **3.3 FIELD PROCEDURES**

The work approach for closure field activities includes:

- Pre-mobilization activities, such as laboratory coordination, clearing the sampling area of obstructions, and preparation of sampling templates for non-porous surfaces;
- Mobilization to the site, grid layouts and sampling of porous, non-porous, and ground surface surfaces and the dry wells;
- Submittal of samples for analysis under standard Chain-of-Custody protocol to the laboratory.

### **3.3.1 Pre-Mobilization Activities**

The primary focus of pre-mobilization activities will be to ensure safe site access, control access to potentially contaminated areas, ensure all inventory is removed, and provide for the health and safety of personnel and the surrounding area before the start of closure activities. The components of pre-mobilization are discussed in the following sections.

#### ***3.3.1.1 Permitting***

VES anticipates that no permits will be required during closure activities; however, VES will provide the U.S. Environmental Protection Agency (USEPA) with a closure activity schedule. In addition, all PCB inventory will be removed from the facility prior to the commencement of sampling activities.

#### ***3.3.1.2 Site-Specific Health and Safety Plan (HASP)***

A written HASP is required for potential hazardous waste investigations and remediation according to the Occupational Safety and Health Administration (OSHA), Title 29 Code of Federal Regulations Part 1910.120(b). A site-specific HASP has been prepared to govern the field activities at the site and is included as an Appendix to the Closure Plan. Field personnel will have a thorough understanding of the HASP prior to mobilizing to the site for sampling activities.

#### ***3.3.1.3 Utility Location***

Although sampling of building interior surfaces is not expected to affect subsurface utilities, VES will flag utilities in the building interior to allow samplers to avoid electrical conduit and water, sewer, natural gas piping within the building. In addition, VES will have all exterior utilities delineated prior to any sampling of the ground surfaces.

### **3.3.2 Porous and Non-Porous Sampling**

#### ***3.3.2.1 Sampling Procedures***

During the sample collection activities specified below, samples will be collected in accordance with the provisions of the SOPs for Porous Surface Sampling (Appendix A, Section 9.0), Wipe Sampling for PCBs (Appendix B), and Collection of Soil Samples for Polychlorinated Biphenyls Analysis (Appendix C). If field conditions dictate the necessity for modified locations, an explanation will be provided in the field logbook.

### **3.3.2.2 Sample Analysis**

All samples collected during closure sampling will be evaluated for PCBs using SW-846 Method 8082. Table 3 presents a summary of the analytes, analytical methods, sample containers, field preservation, and maximum analytical holding times for closure samples.

### **3.3.3 Sample Handling, Packaging, and Delivery to the Laboratory**

Samples will be collected, preserved and handled in accordance with Table 3 and the SOP for Sample Management (see Appendix C). The following information must be recorded on a sample label placed on each sample container:

- Project or job number;
- Sample identification number;
- Date and time of sample collection;
- Analyses to be conducted by laboratory; and
- Sampler's initials.

A unique sample identification number (e.g., VES-BLDG2-IN-F-3) will be given to each sample in the field. It will be comprised of the following:

- VES (for Veolia Environmental Solutions);
- A primary location reference
  - BLDG2 = Building 2
  - BLDG3 = Building 3
  - BLDG4 = Building 4
  - OTH = Other (e.g., ground surface or dry well)
- A sample location reference
  - IN-F = inside floor
  - IN-W = inside wall

- IN-D = inside door surface
  - IN-FJ = inside floor, judgmental sample
  - OUT-W = outside wall
  - OUT-D = outside door surface
  - GRND = ground surface
  - DRY1 = numbered dry well
- A sample number (i.e., 1, 2, 3, etc.).

The sampling numbers will be assigned in the field as the samples are collected and noted adjacent to the sample location on the sampling maps (see figures). Field duplicate samples will be numbered in series with the field original samples in order to provide a blind duplicate sample for the laboratory. The associated field original sample for each field duplicate sample will be noted in the field logbook.

In addition to the packing and transporting procedures identified in the SOP for Sample Management, the packaged sample containers will be placed individually in self-sealing plastic bags and stored in a cooler containing wet ice in preparation for delivery to the laboratory. Ice for the sample cooler will be sufficient to maintain the cooler at  $4 \pm 2$  degrees Celsius until delivery at the laboratory. Samples will be delivered to the analytical laboratory as soon as possible after sample collection but no later than the next morning after the day of collection.

### **3.3.4 Field Documentation**

Field documentation will be maintained in accordance with the SOP for Field Activity Records and sample custody will be maintained and documented in accordance with the SOP for Chain-of-Custody Forms (see Appendix C).

A field supervisor, who is responsible for maintaining field documentation, will direct sampling activities. Sampling information will be recorded in a project field logbook and on Chain-of-Custody (COC) forms. The project field logbook will be used to keep a diary of field activities and to record pertinent data that are not included on the chain-of-custody form.

Thus, field documentation will include:

- The project field logbook that records field activities and pertinent data, including general site conditions, daily weather, arrival and departure of field personnel and subcontractors (as applicable), equipment used onsite, equipment problems, handling and disposal of investigation derived waste, and other relevant information;
- Health and safety documentation as required by the Health and Safety Plan (see Section 3.3.1.2);
- Copies of completed COC forms; and
- A photograph log.

Field personnel will request standard turn-around-times (TAT) for sample analyses and EPA Level II laboratory documentation (with both hardcopy and electronic deliverables) on completed COCs.

### **3.3.5 Sampling Equipment Decontamination**

Key elements of the decontamination procedures to be employed during closure include:

- Sampling equipment will be either disposable or decontaminated prior to and following each use;
- Personnel decontamination procedures will follow all OSHA requirements;
- All site workers will be trained in decontamination procedures.

The proposed method of approach assumes that sampling activities will be performed in Level D PPE.

Prior to the initiation of sampling and between each sampling location, all reusable sampling equipment will be washed and cleaned using the decontamination procedure outlined in Appendix A, Section 11.0:

- Wash with a potable water Alconox solution (or equivalent) Rinse with potable water
- Rinse with hexane

Decontamination of equipment is important to prevent cross-contamination between sample locations by any contaminant from the previous sample. Decontamination of equipment will be

verified by visual inspection (there should be no residue remaining on sampling equipment following decontamination).

### **3.3.6 Investigation Derived Waste**

Chip sampling and wipe sampling are not expected to result in the generation of investigation derived waste with the exception of decontamination fluids. Decontamination water will be collected and managed as PCB waste following sampling activities. Used personal protective equipment (PPE) and sampling equipment will be containerized and disposed of off-site as PCB waste.

Any drill cuttings produced as a result of sampling a borehole adjacent to a dry well will be containerized and stored on-site pending analysis. If the analytical results demonstrate that the drill cuttings are not contaminated, the cuttings will be disposed as solid waste. If the analytical results show the cuttings to be contaminated, they will be disposed of off-site as PCB waste. These materials will be managed and stored in VES Building 3 prior to off-site shipment.

## **4.0 ANALYTICAL PROGRAM**

### **4.1 LABORATORY ANALYTICAL PROGRAM REQUIREMENTS**

#### **4.1.1 Analytical Procedures**

Table 3 summarizes the analytes, analytical methods, sample containers, field preservation, and maximum analytical holding times for currently scoped VES Project sampling programs. The analytical laboratory must be licensed by the ADHS for the analytical method used. Substitutions in analytical methods will be deemed acceptable if an alternative method is included in the ADHS license, attains the required analytical reporting limits, and adequately quantitates the analytes in the samples evaluated.

#### **4.1.2 Required Analytical Reporting Limits**

The analytical laboratory must be able to demonstrate that method reporting limits for all analytes evaluated in a clean matrix (i.e. method or preparation blanks) are no more than half of the applicable regulatory limit of 1 ppm for porous surfaces and soils and 10 micrograms per 100 square centimeters for non-porous materials. The regulatory thresholds and maximum laboratory reporting limits are presented in Table 3. It is recognized that site-specific matrices may have an adverse effect on reporting limits achievable on environmental samples and that these reporting limits may not always be achievable on site sample analyses.

#### **4.1.3 Laboratory Data Reduction and Verification**

The laboratory will perform in-house analytical data reduction and QA review to ensure proper attainment of laboratory quality control criteria prior to producing a final laboratory report. Data reduction, QA review and reporting by the laboratory will be conducted in accordance with the laboratory Quality Assurance Plan and/or the specified methods. A copy of the laboratory Quality Assurance Manual (QAPP) is provided as Appendix E.

#### **4.1.4 Laboratory Reporting**

The laboratory will report data in a sample delivery group of up to 20 associated samples. The SDG will be comprised of those samples designated by field sampling personnel on the Chain-of-Custody forms. The analytical data package will be compliant with Level II data verification requirements including:

- A Case Narrative summary of all analytical methods used for prep/digestion through analysis, including specific identification of instruments used, discussion of factors affecting the analysis and corrective actions taken; justification for dilution(s) of all samples and/or digestates; summary of the source and reasons for variance from the original analytical request; and report of inconsistencies and/or problems with paperwork, shipping and packaging of samples;
- Analytical data results for all field samples with method reporting limit information (*all solid sample results must be reported on dry weight basis*);
- Sample holding times (preparation and analysis dates and times),
- Quality control sample results including those for method blanks, check samples (as applicable), matrix spike/matrix spike duplicate and duplicate samples (as applicable), laboratory control samples; and
- Chain-of-Custody record.

The laboratory will retain full analytical and QC documentation for the sample analyses performed for a period of at least five years. If data verification indicates further review is required, a more complete data reporting package will be requested.

#### **4.2 INDEPENDENT DATA VERIFICATION**

A second level of review of all analytical data and field procedures produced under this SAP will be performed by an entity independent of the laboratory. The data review will consist of an evaluation of:

- Problems with analyses noted in the Case Narrative.
- Review of compliance with holding time limits specified in the analytical methods and/or Table 3 of this SAP. Guidance from USEPA's *Functional Guidelines* (USEPA, 2008) will be utilized in qualifying associated data based on holding time limit exceedances.
- Analytical accuracy will be evaluated by comparing spike sample recoveries and laboratory control sample analyses against QC acceptance criteria specified in Appendix D of this SAP. Guidance from USEPA's *Functional Guidelines* (USEPA, 2008) will be utilized in qualifying associated data based on spike recovery results.

- Analytical precision will be evaluated by comparing matrix duplicate results against the QC acceptance criteria specified in Appendix D of this SAP. Guidance from USEPA's *Functional Guidelines* (USEPA, 2008) will be utilized in qualifying associated data based on precision of analytical results.
- Precision of field sampling and laboratory analysis will be evaluated by comparing blind field duplicate analyses against the QC acceptance criteria specified in Appendix D of this SAP. Associated data will be qualified based on the QA requirements presented in Appendix D.
- Field and laboratory contamination will be evaluated by comparing the results of method or preparation blanks with the acceptance criteria specified in USEPA's *Functional Guidelines* (USEPA, 2008). Guidance from USEPA's *Functional Guidelines* (USEPA, 2008) will be utilized in qualifying associated data based on blank analysis results.
- Field contamination will be evaluated by reviewing the results of equipment rinsate samples. No analytes should be present at concentrations exceeding reporting limits in equipment rinsate samples.

Calculation of data quality indicators during the independent data verification described above will be completed as described in Appendix D.

The review will also assess the correctness and completeness of the data. Further, the quality of the work will be evaluated based on an established set of laboratory guidelines and method requirements. This evaluation will assess whether:

- Sample preparation information is correct and complete;
- Analysis information is correct and complete;
- The appropriate SOPs have been followed;
- Analytical results are correct and complete;
- QC samples and blanks are within established QC limits as calculated using the methods specified in Appendix D of this SAP;
- Any appropriate corrective actions have been implemented and documented; and

- Documentation is complete (all anomalies in the preparation and analysis have been documented; out-of-control incidences are documented, missed holding times and/or reanalysis are explained and documented).

The data verification process will be documented in a Data Verification Memorandum. If quality control or assurance issues are identified in the verification process, an assessment of whether full validation of analytical data is warranted will be conducted and presented in the Data Verification Memorandum.

## **5.0 EVALUATION OF COLLECTED DATA**

### **5.1 SITE CHARACTERIZATION DATA ANALYSIS**

Analytical data will be compared to the regulatory thresholds presented in Table 1 under “Step 6: Specify Performance or Acceptance Criteria.”

### **5.2 SITE CHARACTERIZATION REPORTING**

A report documenting closure activities will be prepared. The report will include a discussion of site conditions; the rationale for sampling locations and sampling procedures; tabulated analytical methods and results; data assessment and interpretation; and conclusions. The report will include a vicinity map, a site map showing the site boundaries and sampling locations, and site photographs. The reports will be sealed and signed by a registered professional engineer.

## 6.0 REFERENCES

- U.S. National Archives and Records Administration. 2009. Code of Federal Regulations Title 40, Part 761.65(e).
- U.S. National Archives and Records Administration. 2009. Code of Federal Regulations Title 40, Part 761, Subpart G.
- U.S. National Archives and Records Administration. 2012. Code of Federal Regulations Title 29 Part 1910.120(b).
- United States Environmental Protection Agency (USEPA). 1986. *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods*. Chapter 9–Sampling Plan. Revision 0. September.
- \_\_\_\_\_, 1991. *Wipe Sampling and Double Wash/Rinse Cleanup as Recommended by the Environmental Protection Agency PCB Spill Cleanup Policy* June 23, 1987, Revised April 18, 1991.
- \_\_\_\_\_, 2008. *National Functional Guidelines for Superfund Organic Methods Data Review*, USEPA-540-R-08-1, June.
- \_\_\_\_\_, 2011. *Standard Operating Procedure for Sampling Porous Surfaces for Polychlorinated Biphenyls (PCBs)*, SDMS DocID 484692, May.

**TABLE 1  
DATA QUALITY OBJECTIVES**

**TSCA Closure – Veolia Environmental Solutions, Phoenix, AZ**

<p>Step 1: State the Problem</p> <p>A. Storage, handling, and processing of material containing PCBs during normal operations within Buildings 2, 3, and 4 may have resulted in PCB impact to the interior surfaces of the buildings.</p> <p>B. Delivery and handling of material containing PCBs during normal operations at loading docks outside Buildings 2, 3, and 4 and the Building 4 exterior wall surrounding the portal located above the loading dock may have resulted in PCB impact to exterior surfaces of the walls.</p> <p>C. Delivery and handling of material containing PCBs during normal operations outside Buildings 2, 3, and 4 may have resulted in PCB impact to ground surfaces adjacent to the buildings including loading docks, asphalt and concrete paved surfaces, and any exposed soils.</p> <p>D. Storage, handling, and processing of material containing PCBs during normal operations conducted at the Buildings 2, 3, and 4 may have resulted in PCB impact to six sealed and the two open dry wells located in proximity to Buildings 2, 3, and 4.</p>
<p>Step 2: Identify the Goal of the Study</p> <p>A. The goal of the study is to demonstrate that interior surfaces of Buildings 2, 3, and 4 meet clean closure requirements for unrestricted access following cleaning procedures.</p> <p>B. The goal of the study is to demonstrate that loading dock walls at Buildings 2, 3, and 4 meet clean closure requirements for unrestricted access following cleaning procedures in addition to the Building 4 exterior wall surrounding the portal located above the loading dock.</p> <p>C. The goal of the study is to demonstrate that ground surfaces adjacent to Buildings 2, 3, and 4 meet clean closure requirements for unrestricted access following any necessary remediation activities.</p> <p>D. The goal of the study is to demonstrate that the dry wells located in proximity to Buildings 2, 3, and 4 meet clean closure requirements for unrestricted access following any necessary remediation activities.</p>
<p>Step 3: Identify Information Inputs</p> <p>A. Interior Building Surfaces</p> <ul style="list-style-type: none"> <li>• Background information on historical operations Regulatory requirements and guidance per 40 CFR 761, Subpart G</li> <li>• PCB concentrations in concrete floors and block walls (porous materials) and on doors and ventilation fan blades (non-porous surfaces) and in accumulated dust above processing areas</li> </ul> <p>B. Loading Dock Wall Surfaces</p> <ul style="list-style-type: none"> <li>• Background information on historical operations</li> <li>• Regulatory requirements and guidance per 40 CFR 761, Subpart G</li> <li>• PCB concentrations in concrete and masonry walls (porous materials)</li> </ul> <p>C. Ground surfaces</p> <ul style="list-style-type: none"> <li>• Background information on historical operations</li> <li>• Regulatory requirements and guidance per 40 CFR 761, Subpart G</li> <li>• PCB concentrations in ground surface materials</li> </ul> <p>D. Dry Wells</p> <ul style="list-style-type: none"> <li>• Background information on historical operations</li> <li>• Regulatory requirements and guidance per 40 CFR 761, Subpart G</li> <li>• PCB concentrations in dry wells, with additional borehole sampling required should the dry well</li> </ul>

sediment concentrations exceed 1 ppm

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

##### A. Interior Building Surfaces

- The boundary of study area will be confined to the portion of the buildings in which PCB transfer, processing, and storage occurred. The study area will consist of the delineated portions of the building floors adjacent walls up to a height of ten feet.
- The study area will include high traffic pathways at the boundary of the study area defined above. In this case, the study area includes doorways into and out of the building and into other rooms or non-PCB handling areas.
- The study area will include areas where dust accumulates above processing areas.
- This study is being conducted for facility closure purposes in accordance with 40 CFR 761, 65(e).

##### B. Loading Dock Wall Surfaces

- The boundary of study area will be confined to the walls of the loading docks where PCB material delivery operations occurred. The study area will consist of the walls of the loading docks, as well as the Building 4 exterior wall surrounding the portal located above the loading dock.
- This study is being conducted for facility closure purposes in accordance with 40 CFR 761, 65(e).

##### C. Ground Surfaces

- The boundary of study area will be confined to the ground surfaces between Buildings 2 and 3, that extending from Building 4 up to 25 feet to the east of the building, ground surfaces extending north of the three buildings to the facility boundary, and ground surfaces extending south of the delivery areas incorporating the ground surfaces surrounding the two open dry wells located there.
- This study is being conducted for facility closure purposes in accordance with 40 CFR 761, 65(e).

##### D. Eight Dry wells

- The boundary of study area will be confined to the eight dry wells, three of which are located within the foot prints of the loading docks located adjacent to Buildings 2, 3, and 4, three of which are located within 30 ft of the corners of these same three buildings, and two located just south of the delivery areas between Buildings 2 and 3 and to the east of Building 4.
- This study is being conducted for facility closure purposes in accordance with 40 CFR 761, 65(e).

#### Step 5: Develop the Analytic Approach

Sample patterns developed with Visual Sample Plan (VSP, Version 6.0) to identify an elliptical hot spot with a semi-major axis of four feet<sup>1</sup> and a width ratio of 0.8 with a 95 percent probability of identifying the hot spot and assuming no false negative errors.

<sup>1</sup> The semi-axis length of 4 feet was selected through an iterative process for Building 4. The starting point of the iterative process was based on Subpart G "PCB Spill Cleanup Policy" in which the sampling requirements for post-spill cleanup as defined in 761.130 were applied to confirmation sampling for closure. Although this section is not directly applicable to closure, it was considered a reasonable starting point in determining the appropriate number and spacing of samples. Section 761.130 specifies that the number of samples for post cleanup spill sampling "should be sufficient to ensure that areas of contamination of a radius of 2 feet or more within the sampling area will be detected, except that the minimum number of samples is 3 and the maximum number of samples is 40." Therefore, the starting point of the iterative process

For high traffic pathways at the boundary of the study area, judgmental samples will be used. Tolerable limits on the decision errors cannot be statistically defined for the judgmental samples collected in high traffic areas.

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

##### A. Interior Building Surfaces

If total PCB concentrations in chip samples (porous media) or wipe samples (non-porous media) are greater than applicable cleanup standards (1 ppm for porous materials and 10 micrograms per 100 square centimeters for non-porous surfaces), the results support a conclusion that the cleaning activities were not adequate to meet clean closure for the interiors of the buildings.

##### B. Loading Dock Wall Surfaces

If total PCB concentrations in chip samples (porous media) or wipe samples (non-porous media) are greater than applicable cleanup standards (1 ppm for porous materials and 10 micrograms per 100 square centimeters for non-porous surfaces), the results support a conclusion that the cleaning activities were not adequate to meet clean closure for exteriors of the buildings.

##### C. Ground Surfaces

If total PCB concentrations in chip samples (porous media) are greater than applicable standards (1 ppm), the results support a conclusion that the cleaning or remediation activities were not adequate to meet clean closure for the ground surfaces adjacent to the buildings.

##### D. Eight Dry Wells

If total PCB concentrations in dry well sediments are greater than applicable standards (1 ppm) and boring samples collected within 5 feet of a given dry well locations, the results support a conclusion that the dry well does not meet requirements for clean closure and additional remediation may be required.

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

##### A. Interior Building Surfaces

- For Building 4, the use of a 95% probability of identifying the hot spot and assuming no false negatives initially resulted in a sampling pattern requiring 117 samples. URS evaluated a lower probability (90%) which reduced the required number of samples to 109. The resultant reduction in the number of samples is not sufficient to justify use of the 90% probability value. Therefore, a 95% probability will be used for all building interior surfaces.
- Discrete chip samples (for porous surfaces) or wipe samples (for non-porous surfaces) collected at each grid point of a triangular grid with a distance of approximately 6.7 feet between grid points. Duplicate samples will be collected at a frequency of 10 percent.
- Biased discrete chip samples collected in:
  - i. Doorways (one sample per man doorway for each doors + two samples per overhead doorway for each doors)
  - ii. One duplicate sample will be collected per building.

was determined to be an ellipse with a semi-major axis of two feet. For the area, in order to have a 95 percent probability of locating an elliptical hot spot with a semi-major axis of 2 feet and a height to width ratio of 0.8 and with zero false negatives, the total number of samples required was estimated to be 478 samples. This collection and analysis of this number of samples is considered to be impractical. Therefore, the ellipse semi-major axis was increased to 3 feet, which resulted in a requirement of 213 samples. Again, this number of samples is considered to be impractical. The ellipse semi-major axis was then increased to 4 feet, which resulted in a requirement of 117 samples. The collection of 117 samples was considered to be feasible and maintains the ability to identify an elliptical hot spot with a semi-major axis of 4 feet with a 95 percent probability.

B. Loading Dock Wall Surfaces

- A 95% probability will be used for the defined loading dock wall surfaces.
- A minimum of three judgmental samples will be collected from the Building 4 exterior wall surrounding the portal located above the loading dock.
- Discrete chip samples (for porous surfaces) or wipe samples (for non-porous surfaces) collected at each grid point of a triangular grid with a distance of approximately 6.7 feet between grid points. Duplicate samples will be collected at a frequency of 10 percent.

C. Ground Surfaces

- A 95% probability will be used for the defined ground surfaces.
- Discrete chip samples (for porous surfaces) collected at each grid point of a triangular grid with a distance of approximately 6.7 feet between grid points. Duplicate samples will be collected at a frequency of 10 percent.

D. Eight Dry wells

- Three sediment samples will be collected from the settling chamber of each dry well. One duplicate per dry well.
- If the PCB concentration for sediment collected from an individual dry well exceeds 1 ppm, samples will be collected from a boring located 5 feet from the dry well with soil samples collected every five feet and at any distinct lithology changes starting at the depth of the bottom of the settling chamber and continuing to a total depth of at least 10 feet below the bottom of the drywell injection pipe. One duplicate per dry well.

**TABLE 2  
TYPE AND NUMBER OF SAMPLES TO BE COLLECTED  
TSCA Closure – Veolia Environmental Solutions, Phoenix, AZ**

<b>Sample Location</b>	<b>Type of Sample*</b>	<b>Number of Field Originals</b>	<b>Number of Field Duplicates</b>	<b>Total Number of Field Samples</b>
<b>Building 2 - Interior</b>				
Floor Sample – Grid	Porous – Chip Sample	166	17	183
Wall Sample – Grid	Porous – Chip Sample	93	10	103
Wall Sample – Grid	Non-Porous – Wipe Sample	8	1	9
Floor Sample – Judgmental	Porous – Chip Sample	9	1	10
Ventilation Fans – Judgmental	Non-Porous – Wipe Sample	4	1	5
Dust Sample – Judgmental	Grab Sample	1	1	2
<b>TOTAL</b>		<b>281</b>	<b>31</b>	<b>312</b>
<b>Building 3 – Interior</b>				
Floor Sample – Grid	Porous – Chip Sample	175	18	193
Wall Sample – Grid	Porous – Chip Sample	96	10	106
Wall Sample – Grid	Non-Porous – Wipe Sample	8	1	9
Floor Sample – Judgmental	Porous – Chip Sample	7	1	8
Ventilation Fans - Judgmental	Non-Porous – Wipe Sample	3	1	4
Dust Sample - Judgmental	Grab Sample	1	1	2
<b>TOTAL</b>		<b>290</b>	<b>32</b>	<b>322</b>

**TABLE 2  
TYPE AND NUMBER OF SAMPLES TO BE COLLECTED  
TSCA Closure – Veolia Environmental Solutions, Phoenix, AZ**

Sample Location	Type of Sample*	Number of Field Originals	Number of Field Duplicates	Total Number of Field Samples
Building 4 - Interior				
Floor Sample - Grid	Porous – Chip Sample	90	9	99
Wall Sample – Grid	Porous – Chip Sample	23	2	25
Wall Sample – Grid	Non-Porous – Wipe Sample	4	1	5
Floor Sample - Judgmental	Porous – Chip Sample	7	1	8
TOTAL		124	13	137
Building 2 –Loading Dock				
Wall Sample – Grid	Porous – Chip Sample	7	1	8
TOTAL		7	1	8
Building 3 – Loading Dock				
Wall Sample – Grid	Porous – Chip Sample	7	1	8
TOTAL		7	1	8
Building 4 – Loading Dock and Exterior Wall Surrounding Portal				
Loading Dock Wall Sample – Grid	Porous – Chip Sample	7	1	8
Exterior Wall Surrounding Portal - Judgmental	Porous – Chip Sample	3	1	4
TOTAL		10	2	12
Ground Surface				
Ground Surface Samples – Grid	Porous – Chip Sample	813	81	894
TOTAL		813	81	894

**TABLE 2  
TYPE AND NUMBER OF SAMPLES TO BE COLLECTED  
TSCA Closure – Veolia Environmental Solutions, Phoenix, AZ**

<b>Sample Location</b>	<b>Type of Sample*</b>	<b>Number of Field Originals</b>	<b>Number of Field Duplicates</b>	<b>Total Number of Field Samples</b>
Eight Dry Wells				
Settling Chambers	Sediments – Grab	24	8	32
Borings (assume one required)	Soils – Grab	3	1	4
<b>TOTAL</b>		<b>27</b>	<b>9</b>	<b>36</b>
Summary				
Building 2 Interior		281	31	312
Building 3 Interior		290	32	322
Building 4 Interior		124	13	137
Building 2 Dock		7	1	8
Building 3 Dock		7	1	8
Building 4 Dock		10	2	12
Ground Surface		813	81	894
Dry Wells		27	9	36
<b>GRAND TOTAL</b>		<b>1,559</b>	<b>170</b>	<b>1,729</b>
*Standard Operating Procedures for Porous Surface Sampling and Wipe Sampling for Non-Porous Surfaces are presented in Appendices A and B.				

**TABLE 3  
SUMMARY OF ANALYTES, ANALYTICAL METHODS, SAMPLE CONTAINERS, PRESERVATION, ANALYTICAL HOLDING TIMES,  
REGULATORY THRESHOLDS, AND REQUIRED LABORATORY REPORTING LIMITS  
TSCA Closure – Building 4, Phoenix, AZ**

Medium	Analyte Group	Analyte(s)	Analytical Method	Sample Container <sup>(1)</sup>	Field Preservation <sup>(2)</sup>	Maximum Analytical Holding Time <sup>(3)</sup>	Regulatory Threshold	Required Laboratory Reporting Limit
Solid	Porous	PCBs	EPA Method 8082	one 2-ounce or 40 mL (at least 10 grams) glass jar with Teflon-lined cap per sample	≤ 6°C	14 days until extraction	1 mg/Kg	0.5 mg/Kg
Solid	Soil/ Sediment	PCBs	EPA Method 8082	one 2-ounce or 40 mL (at least 10 grams) glass jar with Teflon-lined cap per sample	≤ 6°C	14 days until extraction	1 mg/Kg	0.5 mg/Kg
Solid	Non-porous	PCBs	EPA Method 8082	one 500-milliliter glass bottle with Teflon-lined cap per sample <sup>(2)</sup>	Hexane, 4°C	7 days until extractions	10 µg / 100 cm <sup>2</sup>	5 µg / 100 cm <sup>2</sup>

**Notes:**

<sup>(1)</sup> The laboratory will provide all sample containers with appropriate quantities of applicable preservatives.

<sup>(2)</sup> The laboratory will provide an appropriate sample container containing hexane

<sup>(3)</sup> Maximum holding time stated in SDMS DocID 484692 May 2011

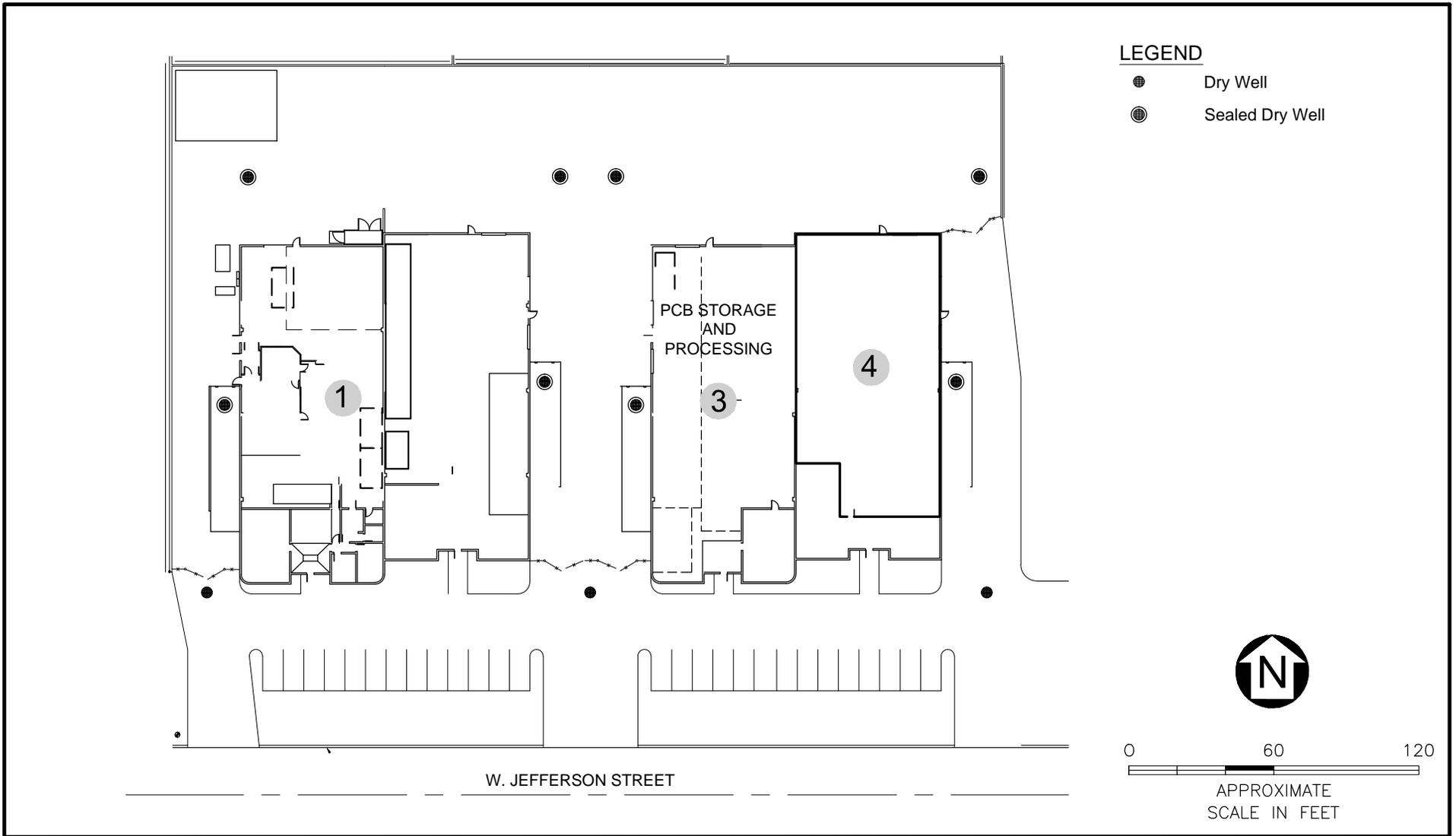
°C = degrees Celcius

mg/Kg = milligrams per kilogram

µg = micrograms

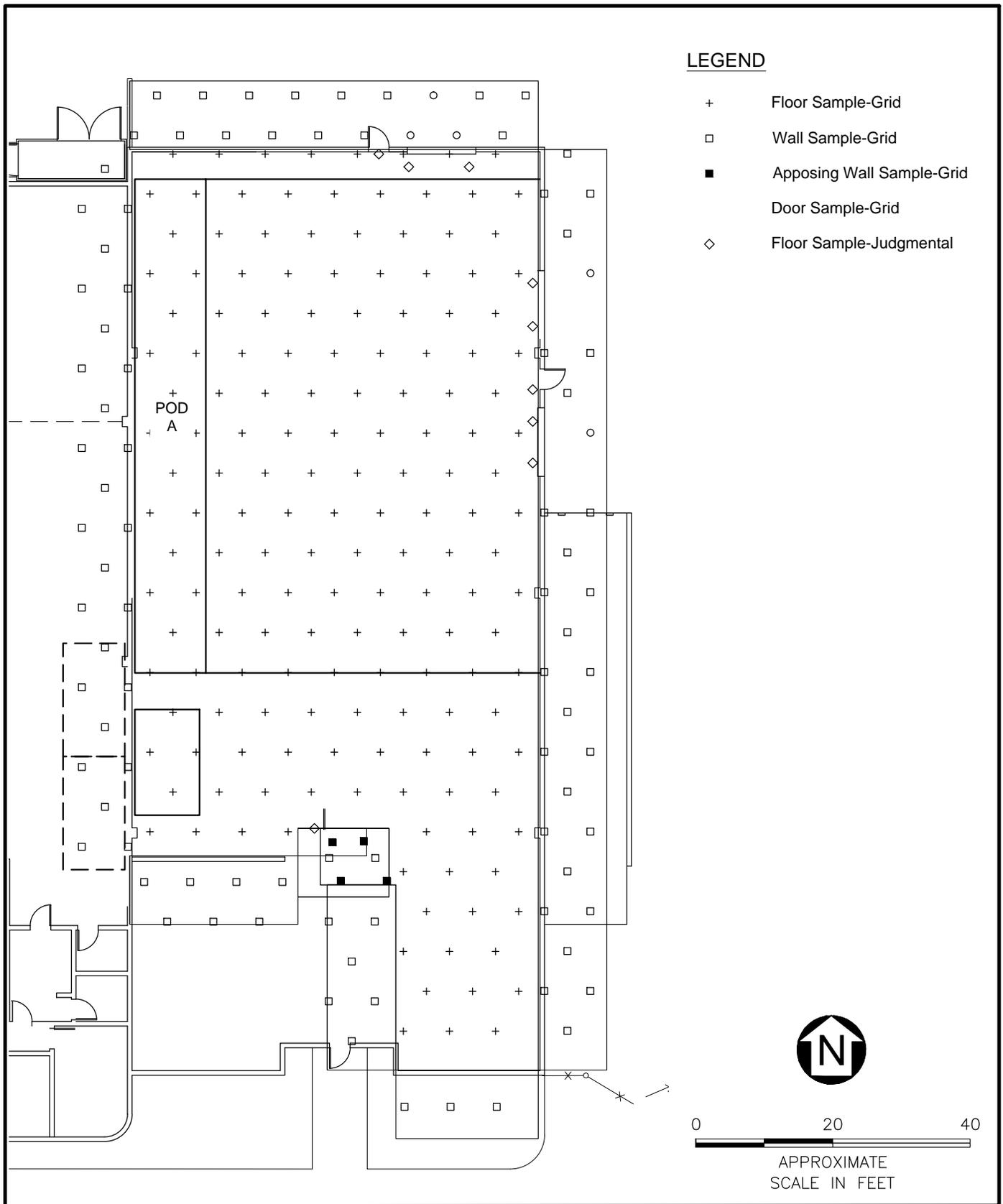
cm<sup>2</sup> = square centimeters

## **FIGURES**

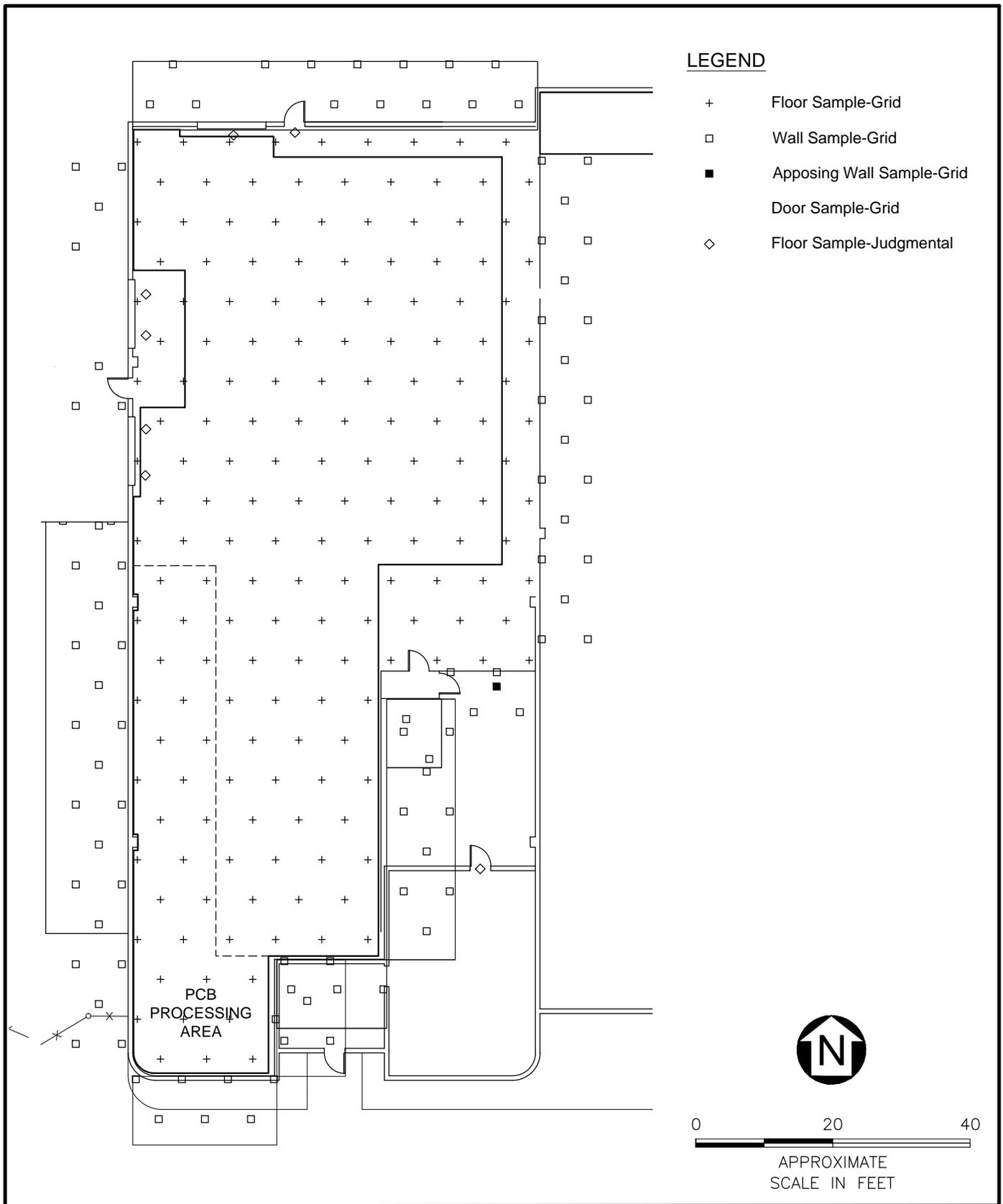


Site Location Map  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ

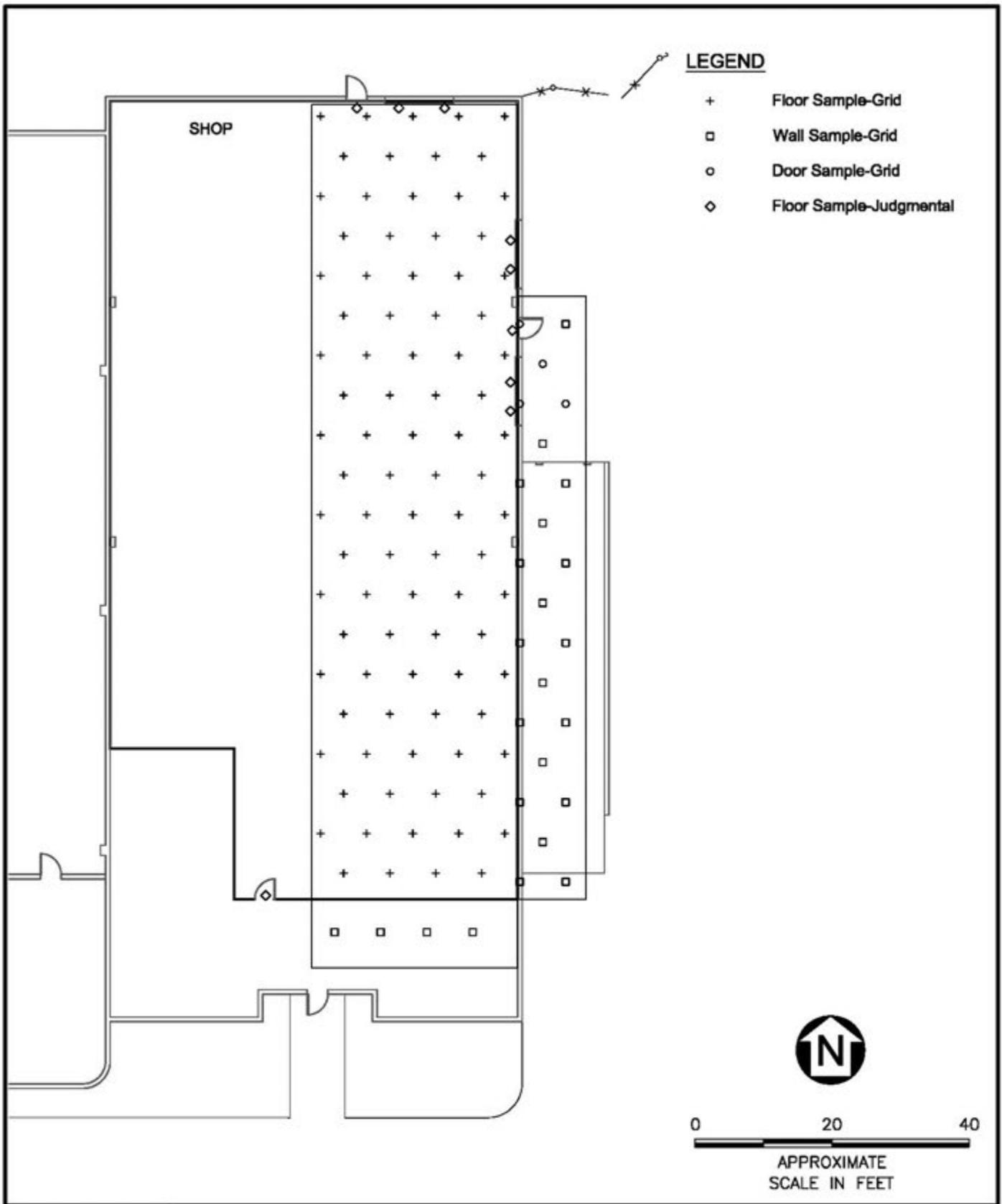




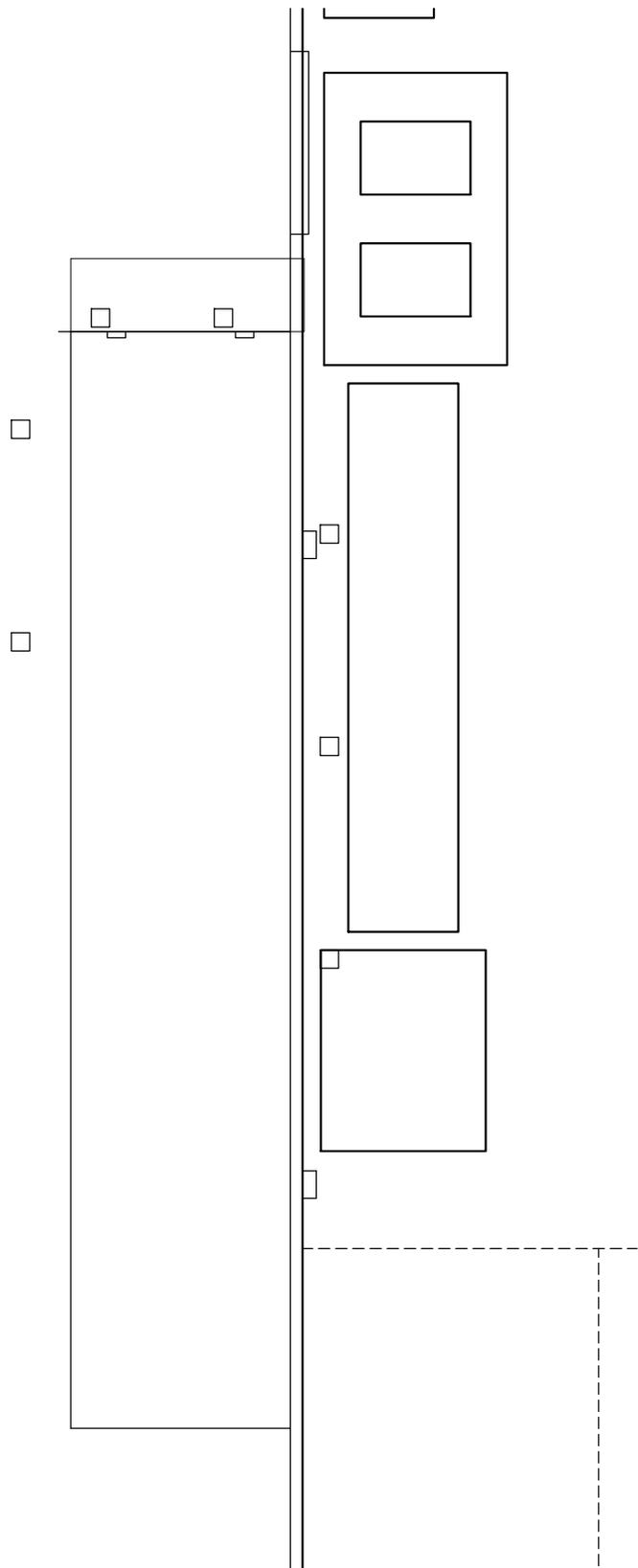
**Building 2 - Proposed Sampling Locations**  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ



**Building 3 - Proposed Sampling Locations**  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ

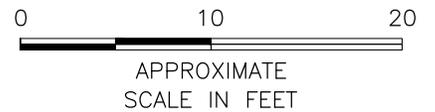


**Building 4 - Proposed Sampling Locations**  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ



**LEGEND**

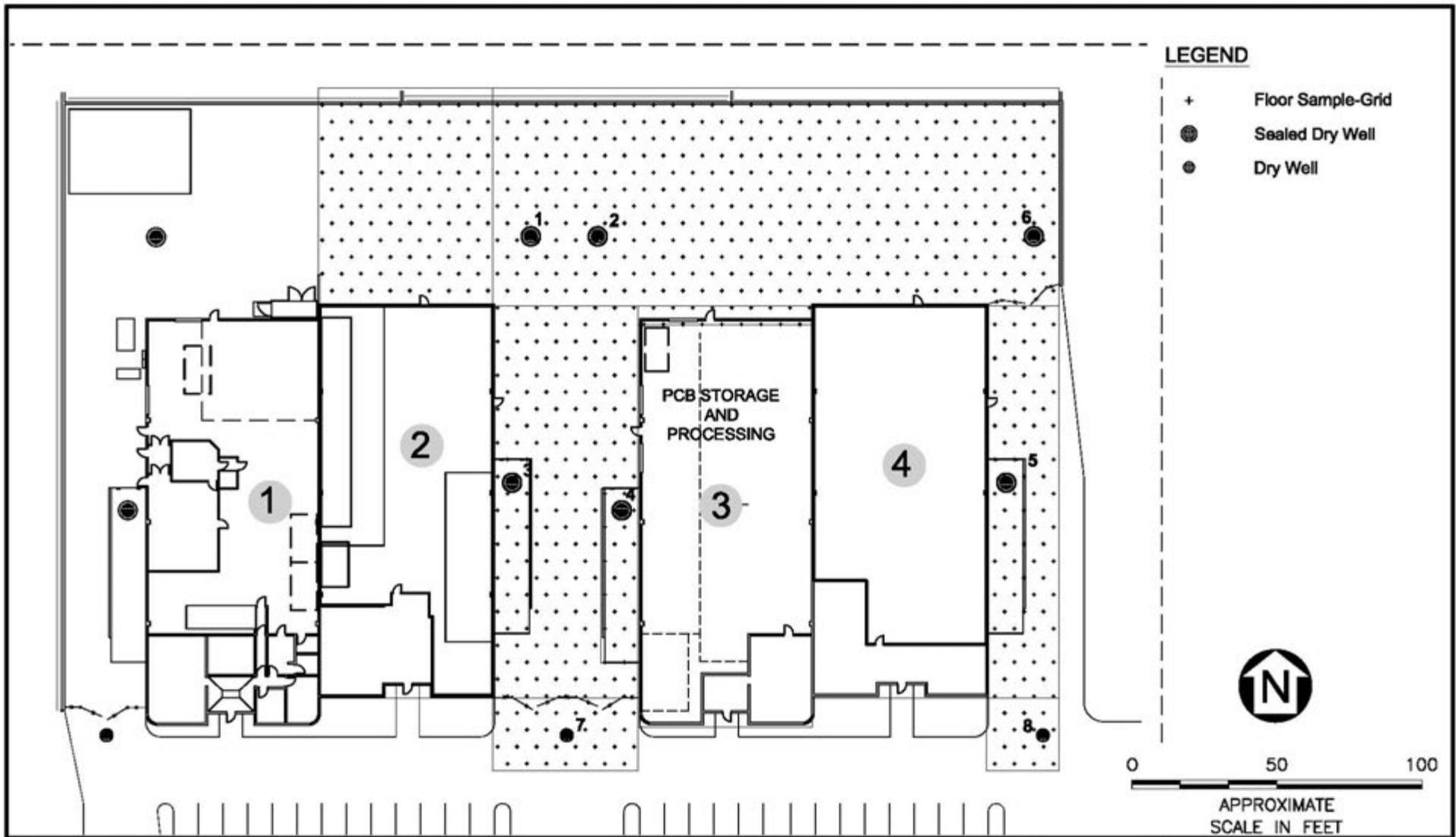
□ Wall Sample-Grid



**Loading Dock Walls - Proposed Sampling Locations**  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ



**Figure 5**



Ground Surface Proposed Sampling Locations  
 Veolia ES Technology Solutions, LLC  
 5736 West Jefferson Street  
 Phoenix, AZ

**APPENDIX A**

**STANDARD OPERATING PROCEDURE FOR SAMPLING POROUS  
SURFACES FOR POLYCHLORINATED BIPHENYLS (PCBS)**

**SDMS DOCID 484692**

UNITED STATES ENVIRONMENTAL PROTECTION AGENCY  
Region 1  
5 Post Office Square, Suite 100  
Boston, MA 02109-3912



**STANDARD OPERATING PROCEDURE FOR SAMPLING POROUS  
SURFACES FOR POLYCHLORINATED BIPHENYLS (PCBs)**

May 2011

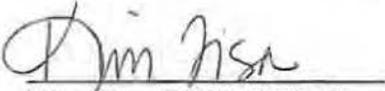


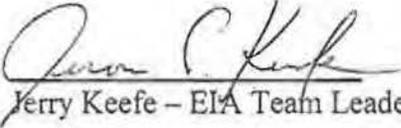
SDMS DocID 484692

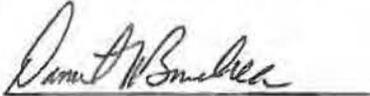
**STANDARD OPERATING PROCEDURE  
FOR SAMPLING POROUS SURFACES  
FOR POLYCHLORINATED BIPHENYLS (PCBs)**

**The Office of Environmental Measurement and Evaluation  
EPA New England – Region 1  
11 Technology Dr.  
North Chelmsford, MA 01863**

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Reviewed by:  5/5/11  
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Reviewed by:  05/23/11  
Jerry Keefe – EIA Team Leader Date

Approved by:  5/23/11  
Dan Boudreau, EIA Chemistry Team Leader Date

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### Attachments:

- Example of Custody Seal and Sample Label
- Example of Chain of Custody Form

## 1.0 Scope and Application

1.1 This Standard Operating Procedure (SOP) is suitable for collection of a porous matrix sample for analysis of Polychlorinated Biphenyls (PCBs).

1.2 This SOP describes sampling techniques for both hard and soft porous surfaces.

1.2.1 Hard surfaces, and most soft surfaces, can be sampled using an impact hammer drill to generate a uniform, finely ground, powder to be extracted and analyzed for PCBs. This procedure is primarily geared at providing enough sample quantity for two analyses. Hard porous surfaces include concrete, brick, asphalt, cement, sandstone, limestone, unglazed ceramics, and other possible PCB suspected material. This procedure may also be used on other softer porous surfaces, such as wood.

1.2.2 Soft surfaces can be sampled using a chisel or sharp knife to generate a representative sample to be extracted and analyzed for PCBs. Soft porous surfaces include wood, wall plasterboard, low density plastics, rubber, caulking, and other PCB suspected material.

1.3 This SOP provides for collection of surface samples (0 – 0.5 inches) and delineation of PCB contamination throughout the core of the porous surface. The procedure can be used to sample the porous surface at distinctly different depth zones.

## 2.0 Method Summary

A one-inch or other sized diameter carbide drill bit is used in a rotary impact hammer drill to generate a fine powder, or other representative sample, suitable for extraction and analysis of PCBs from porous surfaces. This method also allows the use of chisels or knives for the collection of samples from soft porous surfaces for PCB analysis.

## 3.0 Definitions

3.1 Field/Bottle Blank: A sample container of the same lot as the containers used for the environmental samples. This evaluates PCB contamination introduced from the sample container(s) from a common lot.

3.2 Equipment/Rinse/Rinsate Blanks: A sample that is collected by pouring hexane over the sample collection equipment after decontamination and before sample collection. The sample is collected in the appropriate sample container identical to the sample containers. This represents background contamination resulting from the field equipment, sampling procedure, sample container, and shipment.

- 3.3 Field Replicates/Duplicates: Two or more samples collected at the same sampling location. Field replicates should be samples collected side by side. Field replicates represent the precision of the whole method, site heterogeneity, field sampling, and the laboratory analysis.
- 3.4 Field Split Samples: Two or more representative subsamples taken from one environmental sample in the field. Prior to splitting, the environmental sample is homogenized to correct for sample heterogeneity that would adversely impact data comparability. Field split samples are usually analyzed by different laboratories (interlaboratory comparison) or by the same laboratory (intralaboratory comparison). Field splits are used to assess sample handling procedures from field to laboratory and laboratory comparability.
- 3.5 Laboratory Quality Samples: Additional samples that will be collected for the laboratory's quality control program: matrix spike, matrix spike duplicate, laboratory duplicates, etc.
- 3.6 Proficiency Testing (PT)/Performance Evaluation (PE) Sample: A sample, the composition of which is unknown to the laboratory or analyst, provided to the analyst or laboratory to assess the capability to produce results within acceptable criteria. This is optional depending on the data quality objectives. If possible, it is recommended that the PE sample be of similar matrix as the porous surface(s) being sampled.
- 3.7 Porous Surface: Any surface that allows PCBs to penetrate or pass into itself including, but not limited to, paint or coating on metal; corroded metal; fibrous glass or glass wool; unglazed ceramics; ceramics with porous glaze; porous building stone such as sandstone, travertine, limestone, or coral rock; low density plastics such as Styrofoam and low density polyethylene; coated (varnished or painted) or uncoated wood; painted or unpainted concrete or cement; plaster; plasterboard; wallboard; rubber; caulking; fiberboard; chipboard; asphalt; or tar paper.
- 3.8 Shipping Container Temperature Blank: A water sample that is transported to the laboratory to measure the temperature of the samples in the cooler.
- 4.0 Health and Safety**
- 4.1 Eye, respiratory, and hearing protection are required at all times during sample drilling. A properly fitted respirator is required for hard porous surface sampling. A respirator is recommended whenever there is a risk of inhalation of either particulate or volatilized PCBs during sampling.
- 4.2 All proper personal protection clothing and equipment must be worn.

4.3 When working with potentially hazardous materials or situations, follow EPA, OSHA, and specific health or safety procedures.

4.4 Care must be exercised when using an electrical drill and sharp cutting objects.

## 5.0 Interferences and Potential Problems

5.1 This sampling technique produces a finely ground uniform powder, which minimizes the physical matrix effects from variations in the sample consistency (i.e., particle size, uniformity, homogeneity, and surface condition). Matrix spike analysis of a sample is highly recommended to monitor for any matrix related interferences.

5.2 Nitrile gloves are recommended. Latex gloves must not be used due to possible phthalate contamination.

5.3 Interferences may result from using contaminated equipment, solvents, reagents, sample containers, or sampling in a disturbed area. The drill bit must be decontaminated between samples. (see Section 11.0.)

5.4 Cross contamination problems can be eliminated or minimized through the use of dedicated sampling equipment.

## 6.0 Personnel Qualifications

6.1 All field samplers working at hazardous materials/waste sites are required to take a 40 hour health and safety training course prior to engaging in any field activities. Subsequently, an 8 hour refresher health and safety course is required annually.

6.2 The field sampler should be trained by an experienced sampler before initiating this procedure.

6.3 All personnel shall be responsible for complying with all quality assurance/quality control requirements that pertain to their organizational/technical function.

## 7.0 Equipment and Supplies

7.1 This list varies with the matrix and if depth profiling is required

- Rotary impact hammer variable speed drill
- 1-inch or other suitable (1/2, 3/4, etc.) diameter carbide tip drill bits
- Steel chisel or sharp cutting knife, and hammer
- Brush and cloths to clean area
- Stainless steel scoopulas

Aluminum foil to collect the powder sample  
1 quart Cubitainer with the top cut out to collect the powder sample  
Aluminum weighing pans to collect the powder sample  
Cleaned glass container (2 oz or 40 mL) with Teflon lined cap  
Decontamination supplies: hexane, two small buckets, a scrub brush, detergent, deionized water, hexane squirt bottle, and paper towels  
Dedicated vacuum cleaner with a disposable filter or a vacuum pump with a dust filter  
Polyethylene tubing and Pasteur pipettes  
Sample tags/labels, custody seals, and Chain-of-Custody form

## 8.0 Sampling Design

8.1 A sufficient number of samples must be collected to meet the data quality objectives of the project. If the source of the PCB contamination is regulated under the federal TSCA PCB Regulations at 40 CFR Part 761, the sampler should insure that the sampling design is sufficient to meet any investigation or verification sampling requirements. At a minimum, the following is recommended:

- 8.1.1 Suspected stained area (s) should be sampled.
- 8.1.2 At each separate location, collect at least 3 samples of each type of porous surface, regardless of the amount of each type of porous surface present.
- 8.1.3 In areas where PCB equipment was used or where PCBs were stored, samples should be collected at a frequency of 1 sample/100 square feet (ft<sup>2</sup>).

## 9.0 Sample Collection

### 9.1 Hard Porous Surfaces

- 9.1.1 Lock a 1-inch or another size diameter carbide drill bit into the impact hammer drill and plug the drill into an appropriate power source. For easy identification, sample locations may be pre-marked using a marker or paint. (Note: the actual drilling point must not be marked.) Remove any debris with a clean brush or cloth prior to drilling. All sampling decisions of this nature should be noted in the sampling logbook.
- 9.1.2 Use a Cubitainer with the top cut off or aluminum foil to contain the powdered sample. Begin drilling in the designated location. Apply steady even pressure and let the drill do the work. Applying too much pressure will generate excessive heat and dull the drill bit prematurely. The drill will provide a finely ground powder that can be easily collected.

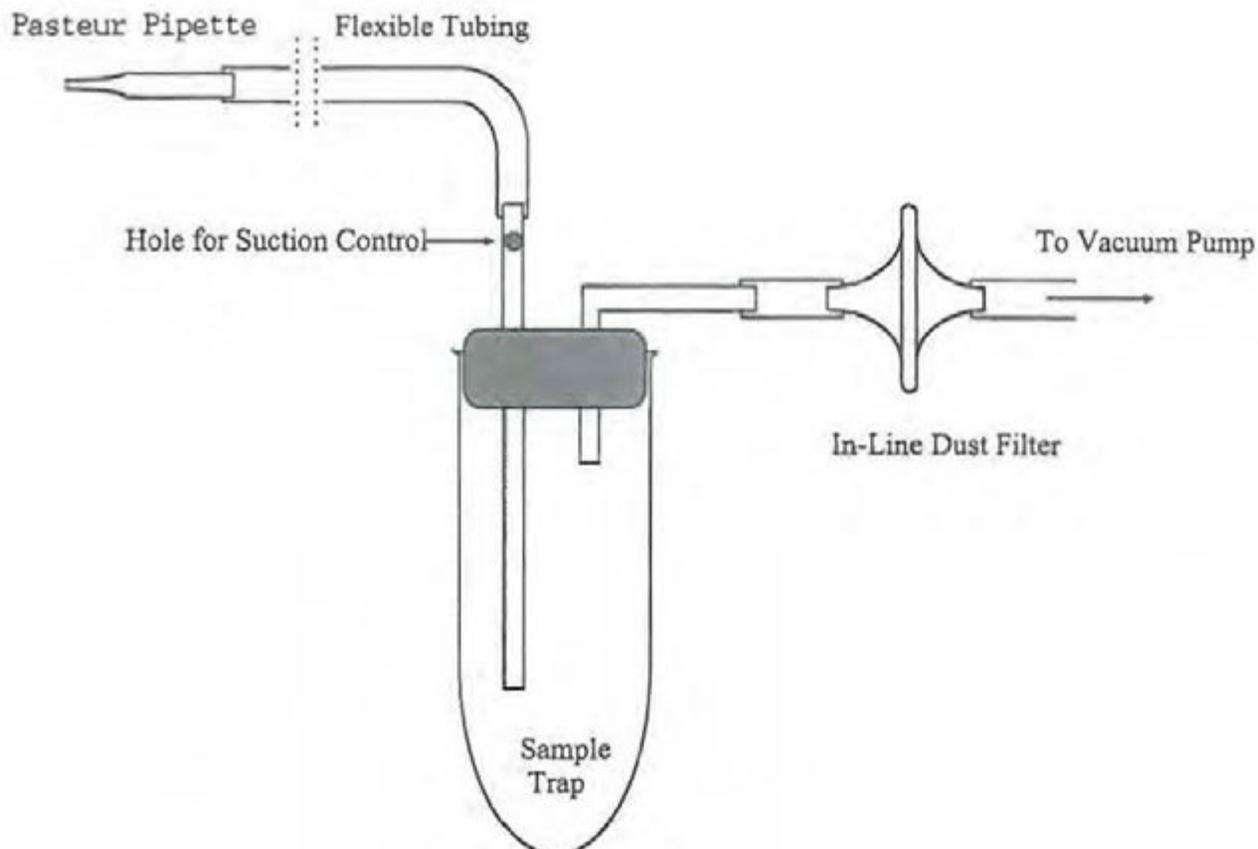
- 9.1.3 Samples should be collected at ½-inch depth intervals. Thus, the initial surface sample should be collected from 0 – 0.5 inches. A ½-inch deep hole generates about 10 grams (20 mL) of powder. Multiple holes located closely adjacent to each other, may be needed to generate sufficient sample volumes for a PCB determination. It is strongly recommended that the analytical laboratory be consulted on the minimum sample size needed for PCB extraction and analysis.
- 9.1.4 **Wall and Ceiling Sampling:** A team of two samplers will be required for wall and ceiling sampling. The second person will hold a clean catch surface (e.g. an aluminum pan) below the drill to collect the falling powder. Alternatively, use the chuck-end of the drill bit and punch a hole through the center of the collection pan. The drill bit is then mounted through the pan and into the drill. For ceilings, the drill may be held at an angle to collect the powder. Thus the driller can be drilling at an angle while the assistant steadies the pan to catch the falling powder. As a precaution, it may be advantageous to tape a piece of plastic around the drill, just below the chuck, to avoid dust contaminating the body of the drill and entering the drill's cooling vents. Caution must be taken to prevent obstruction of the drill's cooling vents.
- 9.2 **Soft Porous Surfaces**
- 9.2.1 The procedure for the hard porous surface may be used for certain soft porous surfaces, such as wood.
- 9.2.2 Samples should be collected at no more than ½-inch depth intervals using a metal chisel or sharp cutting knife. Thus, the initial surface sample should be collected from 0 – 0.5 inches. It is important to collect at least 10 grams for analysis.
- 9.2.3 For soft porous surfaces, such as caulking and rubber, a representative sample can be collected using a metal chisel or sharp cutting knife.
- 9.3 **Multiple Depth Sampling**
- 9.3.1 Multiple Depth Sampling may not be applicable to certain porous surfaces, such as caulking.
- 9.3.2 Collect the surface sample as outlined in Section 9.1 or 9.2.
- 9.3.3 Use the vacuum pump or cleaner to clean out the hole.
- 9.3.4 To collect multiple depths there are two options.

- 9.3.4.1 Option one: drill sequentially ½-inch increments with the 1 inch drill.
- 9.3.4.2 Option two: drill with the 1 inch bit and either make the hole larger or use a smaller bit to take the next ½- inch sample.
- 9.3.5 A stainless steel scoopula will make it easier to collect the sample from the bottom of the hole.

#### 9.4 Vacuum Trap Design and Clean-out

The trap presented in Figure 1 is a convenient and thorough way for collecting and removing concrete powder from drilled holes. The trap system is designed to allow for control of the suction from the vacuum pump and easy trap clean-out between samples. Note, by placing a hole in the inlet tube (see Figure 1), a finger on the hand holding the trap can be used to control the suction at the sampling tip. Thus, when this hole is left completely open, there will be no suction, and the sampler can have complete control over where and what to sample. To change-out between samples the following steps should be taken: 1) the Pasteur pipette and piece of polyethylene tubing at the sample inlet should be replaced with new materials, 2) the portion of the rubber stopper and glass tubing that was in the trap should be wiped down with a clean damp paper towel (wetted with deionized water) and then dried with a fresh paper towel, 3) a clean pipe cleaner should be drawn through the glass inlet tube to remove any concrete dust present, and 4) the glass tube or flask used to collect the sample should be swapped out with a clean decontaminated sample trap. Having several clean tubes or flasks on hand will facilitate change-out between samples.

Figure 1



Note: the holes should be vacuumed thoroughly to minimize any cross-contamination between sample depths and the bits should be decontaminated between samples. (See Section 11.0)

## 10.0 Sample Handling, Preservation, and Storage

- 10.1 Samples must be collected in glass containers for PCB analyses. In general, a 2-ounce sample container with a Teflon-lined cap (wide-mouth jars are preferred) will hold sufficient mass for most analyses. A 2-ounce jar can hold roughly 90 grams of sample.
- 10.2 Samples are to be shipped refrigerated and maintained at  $\leq 6^{\circ}\text{C}$  until the time of extraction and analysis.
- 10.3 The suggested holding time for PCB samples is 14 days to extraction.

## **11.0 Decontamination**

- 11.1 Assemble two decontamination buckets. The first bucket contains a detergent and potable water solution, and the second bucket is for rinsate. Place all used drill bits, hose for the vacuum cleaner, and utensils in the detergent and water bucket. Scrub each piece thoroughly using the scrub brush. Note, the powder does cling to the metal surfaces, so care should be taken during this step, especially with the twists and curves of the drill bits. Next, rinse each piece with water and hexane. Place the rinsed pieces on clean paper towels and individually dry and inspect each piece. Note: all pieces should be dry prior to reuse.
- 11.2 Lightly contaminated drill bits and utensils may be wiped with a hexane soaked cloth and hexane rinsed for decontamination.

## **12.0 Data and Record Management**

- 12.1 All data and information collection should follow a Field Data Management SOP or Quality Assurance Project Plan (QAPP).
- 12.2 Follow the chain of custody procedures to release the samples to the laboratory. A copy is kept with the sampling records.
- 12.3 The field data is stored for at least 3 years.

## **13.0 Quality Control and Quality Assurance**

- 13.1 Representative samples are required. The sampler will evaluate the site specific conditions to assure the sample will be representative.
- 13.2 All sampling equipment must be decontaminated prior to use and between each discrete sample.
- 13.3 All field Quality Control (QC) sample requirements in a Sample and Analysis Plan (SAP) or QAPP must be followed. The SAP or QAPP may involve field blanks, equipment blanks, field duplicates and/or the collection of extra samples for the laboratory's quality control program.
- 13.4 Field duplicates should be collected at a minimum frequency of 1 per 20 samples or 1 per non-related porous matrix, whichever is greater.

## 14.0 Waste Management and Pollution Prevention

- 14.1 During field sampling events there may be PCB and/or hazardous waste produced from the sample collection. The waste must be handled and disposed of in accordance with federal, state, and local regulations. The dust filter, and tubing if a vacuum pump is used, is disposed after each site investigation. This waste will be treated as PCB waste if the samples are positive for PCBs. It may be possible to manage or dispose of the waste produced at the site where the work was performed. If the site does not meet regulatory requirements for these types of activities, the waste must be transported to a facility permitted to manage and/or dispose of the waste.

## 15.0 References

1. Guidance for the Preparation of Standard Operating Procedures for Quality-Related Operations, QA/G-6, EPA/600/R-96/027, November 1995.
2. 40 CFR Part 761 – Polychlorinated Biphenyls (PCBs) Manufacturing, Processing, Distribution In Commerce, and Use Prohibitions
3. Sample Container and Holding Time: RCRA SW 846, Chapter 4, Table 4.1, Revision 4, February, 2007.

### Example of Sample Label and Custody Seal

<b>U.S. ENVIRONMENTAL PROTECTION AGENCY – REGION I BOSTON, MASS.</b>	
<b>LABEL</b>	NAME OF UNIT AND ADDRESS  ENVIRONMENTAL SERVICES DIVISION 60 WESTVIEW STREET LEXINGTON, MASSACHUSETTS 02173
	DATE: YR/MO/DAY  TIME  STATION NO.
<b>SAMPLE</b>	SOURCE OF SAMPLE  SAMPLING CREW (FIRST, INITIAL, LAST NAME)
	SAMPLE NO. SUB NO.
	PRESERVATIVE  AMOUNT
	ANALYSIS

 UNITED STATES ENVIRONMENTAL PROTECTION AGENCY OFFICIAL SAMPLE SEAL	SAMPLE NO.	DATE	SEAL BROKEN BY  DATE	EPA FORM 7500-2 (07-75)
	SIGNATURE			
	PRINT NAME AND TITLE <i>(Inspector, Analyst or Technician)</i>			



**APPENDIX B**

**WIPE SAMPLING AND DOUBLE WASH/RINSE CLEANUP AS  
RECOMMENDED BY THE ENVIRONMENTAL PROTECTION AGENCY  
PCB SPILL CLEANUP POLICY**

WIPE SAMPLING AND DOUBLE WASH/RINSE CLEANUP  
AS RECOMMENDED BY  
THE ENVIRONMENTAL PROTECTION AGENCY PCB SPILL CLEANUP POLICY

June 23, 1987

Revised and Clarified on April 18, 1991

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## **I. WIPE SAMPLING ACCORDING TO THE PCB SPILL CLEANUP POLICY**

### Introduction:

This document was prepared following the publication of the PCB Spill Cleanup Policy in the Federal Register on April 2, 1987. The procedures were demonstrated by EPA PCB program technical staff at PCB Forum '87 and PCB Forum '88. These PCB forums were privately sponsored seminars discussing the requirements of the recently issued PCB Spill Cleanup Policy. The seminars were publicly announced and held in eight cities near the EPA Regional Offices.

The revisions and clarifications to the document include the addition of an Introduction heading, the addition of three paragraphs to the Background heading, and the amendment to item 4 in "An Example of a Wipe Sampling Procedure."

This document was revised and clarified because it did not clearly and completely state EPA's intentions in an area where details were essential, that is the original version of this document assumed that a gloved hand would apply the gauze with moderate pressure, but inadvertently this requirement was never explicitly stated in the example of the wipe sampling procedure. The gloved-hand application of the gauze might have been assumed since the gloves were to be discarded after each sample. The procedure clearly did not say to apply the gauze to the surface with forceps. The EPA demonstrations and discussions at the PCB Forums clearly emphasized the pressurized application of moistened cotton gauze to the surface with a gloved hand.

### Background:

The PCB spill Cleanup Policy requires wipe sampling for the determination of surface levels of PCBs resulting from PCB spills onto hard, "smooth", surfaces such as metal, wood, concrete, plastic, and glass (see Tables 1 and 2). There are several activities surrounding a PCB spill cleanup where wipe sampling may be used: (a) site characterization; (b) interim evaluation of the progress of the cleanup; and (c) the final process to verify that the cleanup has met requirements of the PCB Spill Cleanup Policy.

Wipe sampling has a number of advantages. The most apparent advantage is that wipe sampling is probably the best way to determine smooth "impervious" surface concentrations. Wipe sampling is most effective in areas with relatively large, flat, easily accessible surfaces where an accidental and/or short time

exposure to PCBs has occurred. The surfaces which are sampled by wipe sampling in many cases will have been (or will be) cleaned by wiping or wiping-related activities.

Wipe sampling is best used in conjunction with statistical random sampling and/or area sampling techniques. Reduction in sampling errors for all kinds of sampling procedures can be accomplished by statistical selection of the smaller sampling sites selected to represent a larger area. Non-sampling errors may be reduced by maintaining consistency within the sampling activities; use of comprehensive quality control procedures and samples; and wherever possible, establishing a reference point for comparison.

Unfortunately, wipe sampling is not quantitative because of the fairly large variability in several component parts of sampling and the relative inefficiency of extraction of the analyte of interest from the wipes. Wipe sampling evaluation study results are known to vary widely, for example, when the same sampling is done (1) by different samplers; (2) on similarly contaminated surfaces having different textures or porosities; (3) using no solvent or solvents having different polarities; and (4) using different kinds of wiping material such as filter paper or cotton gauze.

When a decision is made to use wipe sampling, (1) it should be assumed that the results are not always reproducible; (2) extra care should be used to minimize the variability and optimize quantitation; and (3) even if representative sampling is employed, wipe sampling results can indicate residual levels substantially below true surface levels. In developing the PCB Spill Cleanup Policy, EPA has considered the advantages and disadvantages of wipe sampling and accordingly has established allowable residual PCB levels as measured by wipe sampling.

Since the objective of surface sampling is to remove PCB liquids and particles, which may be adhering to the surface, from the surface an aggressive sampling procedure is necessary. The aggressive sampling is appropriate since often the surfaces being sampled have been aggressively cleaned and may drive residual PCBs into the surface. For determining the PCB surface concentrations on smooth surfaces, EPA recommends wipe sampling using cotton gauze as the wipe medium and using a gloved or doubly gloved hand to apply the wipe to the surface. This procedure requires changing into new/clean gloves between samples. EPA recognizes that there may be some transport of PCBs from the gauze to the surface of the gloves. However, this potential loss is considered more acceptable than the problems from the disadvantages of other wipe sampling procedures.

Procedures employing filter paper and/or glass fiber pads and application of these pads to surfaces by swabbing, dipping, or brushing with a pair of forceps are unacceptable. EPA

recognizes that this kind of wipe sampling technique may be

widely applied to address other kinds of surface sampling objectives. However, to meet EPA's PCB surface sampling objectives, these procedures are less efficient and less effective than hand wiping with the more absorbent cotton gauze.

Any compositing of wipe samples or sampling of areas larger than 100 cm<sup>2</sup> may not address the intent of PCB Spill Cleanup Policy verification sampling.

### **Answers to Questions on Wipe Sampling Procedures:**

#### **Why is does it take so much care to wipe sample correctly?**

There is a considerable variability possible among wipe sampling results due to (a) the sampling technique of the sampler and (b) the efficiencies of removing PCBs from several matrices and placing the PCBs into several other matrices. Therefore it is important to reduce this variability to the maximum extent possible, so that in the event of a verification analysis by quality control samplers or government enforcement inspectors, similar wipe sampling results will be obtained for a clean site.

Two factors increase the probability of reducing errors introduced by the sampler's technique: consistency and quality control. Consistency is aided by proper training, easily understood sampling procedures, immediate availability of proper supplies, and whenever possible, using the same sampler to do all sampling at a particular site. Quality control procedures provide reference points and comparisons for the field sample results. When the analytical results from quality control samples indicate potential sampling and analysis problems, there is often sufficient time to reexamine field results. Quality control sampling can reduce or eliminate additional sampling and analysis start up and/or additional cleanup costs.

The reproducibility and efficiency of transferring residual PCBs from one place to another require that such residual PCBs must have a much greater affinity to partition, in one or more steps, from the place of origin to the ultimate destination. For all transfer steps, PCBs must exhibit a much greater propensity to be in the destination medium than in the medium of origin. There are several transfer steps in the process which starts from the removal of PCBs from the surface sampled and ends with the production of a PCB surface concentration by way of instrumental analysis.

The first of these transfer steps is removing residual PCBs from the surface to be sampled and transferring them into the sampling medium\*. Gauze pads are sturdier, allow better surface to surface contact, and absorb more solvent (and more PCBs) than filter paper. Therefore, gauze pads are the absorbent/sampling medium of choice. Since PCBs are very soluble in organic solvents, organic solvent is used to moisten the gauze pads to ease the transport of PCBs from the sampled surface into the sampling media. Once the areas of where the spill occurred have been sampled (after cleanup) and the residual PCBs have been transported to the moistened gauze, then the gauze is air dried and stored/shipped for chemical analysis. The gauze is dried so as to facilitate transfer by organic solvent from the gauze to another medium during the laboratory extraction step.

In the extraction step the PCBs must be isolated from the gauze in a form amenable to the chemical analysis methods to be used. The PCBs now in the gauze are usually extracted into a solvent by repeated rinsing with and subsequent collection of organic solvent. The extraction solvent is removed from the PCBs by evaporation of the solvent prior to chemical analysis. The more volatile organic solvent evaporates and leaves the less volatile PCBs in a more concentrated solution for further treatment or instrumental analysis.

#### **What is the best way to wipe sample for PCBs on smooth surfaces?**

There are several steps in a wipe sampling procedure. The first step is to prepare the sampler for the sampling activity. The sampler may have to be advised of (through a briefing or a refresher course), or trained in, the objectives of the sampling program and the procedures to be used to accomplish those objectives.

Once advised of the objectives and sampling procedures, the sampler must either prepare or obtain the sampling plan and sampling materials. The sampler must know the exact sampling sites or know the exact procedure for selecting those sites. The sampling supplies must be sufficient in quantity and quality for all normally expected occurrences. Provisions should be also made for quality assurance samples, chain of custody forms, and shipping materials for storage.

\* When PCB-contaminated office paper has been solvent rinsed, then wipe sampled and bulk sampled, some recent chemical analysis results indicate that the PCB concentration in the surface wipes is not the same as the concentration in the bulk samples. PCB levels in uncontaminated paper were used as a control. The difference in PCB levels in the wipe samples and bulk samples may

be explained by PCB migration into the paper either during cleanup to remove PCBs or during the wipe sampling step.

An important series of quality assurance measures taken before on-site sampling occurs may save considerable expense from collecting contaminated or unusable wipe samples. Sampler training can include practice sampling of surfaces spiked with PCB surrogate compounds, such as tri- and tetrachlorobenzenes to sharpen skills (a) in wiping thoroughly and consistently, and (b) avoiding cross contamination. In addition, before field sampling is conducted, method blanks can be used to verify that sampling equipment supplies and procedures do not introduce PCBs or analytical interferences to the wipe samples. Complete supplies for sampling should be cleaned, a fraction of the supplies sampled individually or through method blanks, and, if clean, the supplies should be protected against contamination or destruction while being transported to the sampling site and while at the sampling site before actual sampling occurs.

The sampler arrives at a sampling site and determines the exact location where the 100 square centimeter ( $\text{cm}^2$ ) sample will be taken. The sample location may be marked or framed by a template. The sampler must be conscious of possibility of cross contamination during all stages of the sampling activity. All surfaces should be wiped with as uniform a pressure as possible. It is important to use the appropriate pressure to thoroughly wipe materials off the surface. Wiping proceeds from left to right in rows from the top to the bottom of the framed sampling area. The sampling area is wiped again with the same uniform pressure in columns from the top to the bottom from the left side to the right side of the entire framed area. It is not critical whether wiping starts at the top left or with rows first and then columns. The objective is to systematically, thoroughly, and consistently wipe the entire framed area twice, each time from a different direction and orientation.

Once the area has been wiped, the sampling gauze is allowed to air dry and is replaced in the sample vial. The sample vial is then labelled, the chain of custody filled out, and the sample prepared/stored for shipping.

Table 1

**SUMMARY OF CLEANUP LEVELS  
BASED ON THE EPA PCB SPILL CLEANUP POLICY**

**Requirements for Cleanup of Low-Concentration Spills  
Which Involve Less Than One Pound PCBs by Weight  
(Less Than 270 Gallons of Untested Mineral Oil  
[Containing Less Than 500 ppm PCBs])**

Solid Surfaces (except for all indoor, residential surfaces other than vault areas)	Double washed/rinsed
All Indoor, Residential Surfaces Other Than Vault Areas	10 micrograms per 100 cm <sup>2</sup> by standard commercial wipe tests
Soil	Remove visible traces of the spill and soil within a one foot buffer of the visible traces

Table 2

**SUMMARY OF CLEANUP LEVELS  
BASED ON THE EPA PCB SPILL CLEANUP POLICY**

**Requirements for Cleanup of  
High-Concentration Spills and Low-Concentration Spills  
Involving One Pound or More PCBs by Weight  
(270 Gallons or More of Untested Mineral Oil  
[Containing Less Than 500 ppm PCBs])**

Residential/Commercial/Rural

Indoor (except vaults), and Outdoor High Contact	10 micrograms per 100 cm <sup>2</sup>
Indoor Vaults	10 micrograms per 100 cm <sup>2</sup>
Outdoor Low Contact Porous Surface Option	10 micrograms per 100 cm <sup>2</sup> 100 micrograms per 100 cm <sup>2</sup> plus encapsulation
Soil	10 ppm Plus a 10 Inch Cap

Restricted Access (Non-Sub-Station)

High Contact Surfaces	10 micrograms per 100 cm <sup>2</sup>
Low Contact Indoor Surfaces Porous Surface Option	10 micrograms per 100 cm <sup>2</sup> 100 micrograms per 100 cm <sup>2</sup> Plus Encapsulation
Outdoor Low Contact Surfaces	100 micrograms per 100 cm <sup>2</sup>
Soil	25 ppm

Outdoor Electrical Substations

Surfaces	100 micrograms per 100 cm <sup>2</sup>
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Soil

25 ppm or 50 ppm with Notice

**Additional Wipe Sampling Information  
(Contents)**

1. An Example of a List of Wipe Sampling Supplies.
2. An Example of Sample Site Preparations.
3. An Example of a Wipe Sampling Procedure.
4. A Detailed Description of Quality Controls for Wipe Sampling Activities.
5. Wipe Sampling Quality Control Samples (Summary).
6. An Example of Quality Assurance Procedures Useful When Conducting Wipe Sampling Activities.
7. An Example of Procedures to Use When Cleaning Wipe Sampling Equipment.

**An Example of a List of Wipe Sampling Supplies**

Copy of Sampling Procedures and Study Objectives  
Pen (Indelible Ink)  
Pre-numbered Sample Labels  
Tape to Cover Labels  
Chain of Custody Forms  
Screw Top Vials with Teflon Lined Caps  
    These Vials Contain Pre-Cleaned 3" x 3" Surgical Gauze Pads  
Teflon Squirt Bottle for Applying Solvent to Wipes and Washing  
Solvent, preferably in a bottle with a volumetric delivery top  
Graduated cylinder, when not using a volumetric delivery top  
Disposable Gloves  
Metal Ruler  
Sampling Template  
Forceps for Removing (Replacing) Gauze from (into) Vials  
Disposable Wipes (for cleaning ruler)  
Garbage Bags/Containers (for disposal of gloves and solid waste)  
Funnel  
Five Gallon Solvent Can for Disposal of Rinse Solvent  
Shipping/Storage Containers for Samples  
Sampling Site Description Forms with Optional Instant Print  
    Camera

### **An Example of Sample Site Preparations**

At each sample site location:

- Mark the exact sample site with the template or a ruler
  
- If the site is not easily marked with a template or ruler (an irregular non-planar surface), write a detailed description of the area sampled. A instant print photograph with the ruler included (for scale) is a very valuable descriptor.
  
- Prepare all necessary forms and sampling logs for entry of the sampling time, date, location, and other information describing the sampling at that particular site.
  
- Prepare all sampling equipment for sampling the site.

### **An Example of a Wipe Sampling Procedure**

Assume that the exact sampling site has been marked.

1. With gloved hands, remove the cap from the sampling vial.
2. With the forceps, remove the gauze from the sampling vial.
3. From a solvent bottle, use the volumetric delivery device or fill a graduated cylinder with 5 milliliters of solvent to the gauze.
4. Immediately begin applying the gauze using a gloved hand and, applying pressure, wipe the marked area completely twice, from left to right and then from top to bottom.
5. Let the gauze air dry.
6. Fold the dry gauze (sampled side inward) and return it to the sample vial.
7. Cap the sample vial.
8. Remove and discard the gloves.
9. Label the vial and fill out sampling details on the sampling forms.
10. Fill out chain of custody forms and prepare the sample for storage and shipping.

### **A Detailed Description of Quality Controls for Wipe Sampling Activities**

Several kinds of quality control (QC) samples should be used. Each kind of sample provides an indication of the reliability of a part of the sampling and analysis process.

It is better not to identify QC samples as such when submitting the QC samples to the analytical laboratory. It is best to randomly number all samples when submitting them to the analytical laboratory. The chemical analysis laboratory does not need to know sample descriptions except for matrix type or in the event of the presence of an unusually high concentration in the wipe. Specific identification of the QC samples will not be necessary since the concentration range in these samples should be in the normal operating range of the analytical instruments.

Vials refer to the glass vials containing sampling gauze.

1. Field Blanks - at least 5% of the total samples include at least two samples each from the following:
  - a. Ship unopened vials back for analysis.
  - b. With gloved hands, remove the cap from a sample vial for the estimated time (record this time) of normal wipe sampling, allow the gauze to air dry without applying it to any surface, and proceed with step 7 in the wipe sampling procedure.
  - c. Use the wipe sampling procedures to wipe some areas/surfaces near the sampling site but which are not expected to be contaminated.
2. Duplicates - at least 5% of total samples including at a minimum the designated samples from both the following groups:
  - a. Double wipe at least two sample sites, label which was the first wipe and which was the second wipe for each of the two sites, for each kind of surface sampled.
  - b. For at least two sample sites for each kind of surface sampled, wipe two adjacent identical or nearly identical areas. Clearly identify the samples as being adjacent to one another in the sample description forms.

**A Detailed Description of  
Quality Controls for Wipe Sampling Activities  
(Continued)**

3. Field Spikes - at least 5% of total samples including at a minimum the designated samples from each of the following groups for each kind of surface sampled. Clearly describe these samples on the sample description forms.
  - a. For two vials or more, remove each gauze and moisten as for sampling and spike each wet gauze with ten micrograms each of the kind of PCBs which was spilled, wipe a contaminated surface adjacent to a sampled surface as in 2b (above), let the gauze air dry, replace the gauze, and proceed with step 7 in the wipe sampling procedure.
  - b. For a second pair of vials or more, remove each gauze and moisten as for sampling, wipe a contaminated surface adjacent to a sampled surface as in 2b (above), after wipe sampling (but before air drying) spike each wet gauze with ten micrograms each of the kind of PCBs which was spilled, let the gauze air dry, replace the gauze in the vials, and proceed with step 7 in the wipe sampling procedure.
  - c. For a third pair of vials or more, spike sampling surfaces adjacent to another sampled surface as in 2b (above) with ten micrograms each of the kind of PCBs which was spilled and allow to air dry; remove each gauze and moisten as for sampling; wipe the surface; let the gauze air dry, replace the gauze in the vials; and proceed with step 7 in the wipe sampling procedure.

**Wipe Sampling Quality Control Samples (Summary)**

1. Field Blanks - At least two samples from each category
  - a. For each spill site prepare the following blanks:
    - i. Unopened sampling vials containing gauze
    - ii. Remove gauze but do not use to wipe
  - b. For each kind of surface, wipe an uncontaminated 100 cm<sup>2</sup> surface with a gauze as a blank surface
2. Duplicate Samples - At least 5% of total samples
  - a. For each kind of surface at each spill site:
    - i. Double wipe at least two sample sites
    - ii. Side by side wipe at least two sample sites
3. Spiked Samples - At least 5% of total samples
  - a. Wipe no less than two samples each for each kind of surface at each spill site. All are side by side paired samples. One sample for each pair is untreated, for the other sample:
    - i. Spike gauze with 10 micrograms of PCBs, then wipe the 100 cm<sup>2</sup> area
    - ii. Wipe the 100 cm<sup>2</sup> area first, then spike gauze with 10 micrograms of PCBs
    - iii. Spike the 100 cm<sup>2</sup> site with 10 micrograms of PCBs, then wipe

**An Example of Quality Assurance Procedures  
Useful When Conducting Wipe Sampling Activities**

1. Designate a person, not the sampler or chemical analyst, who is responsible for quality assurance and quality control including: training, preparation of sampling supplies, wipe sampling, sample preparation/extraction, chemical analysis, analytical data reduction, reporting of the sampling results, and conclusions drawn from the results.
2. Document the objectives of the wipe sampling and subsequent chemical analysis. Include performance requirements such as number of samples required, precision, accuracy, measurable deliverables, and schedules.
3. Develop a quality assurance plan which includes: the objectives; quality assurance/quality control procedures, audits, and schedules; persons responsible for all aspects of the sampling and chemical analysis efforts; references to all safety, training, sampling, and chemical analysis procedures; and corrective actions (including approximate times before corrective actions will occur) to be taken in the event that documented procedures cannot be or have not been followed.
4. Verify that staff doing sampling are the designated staff or suitably trained and informed replacements for the designated staff.
5. Verify that the sampling equipment and the sample gauze/vials are not going to introduce contamination into the samples.
6. Verify that sufficient quality control samples are taken and taken properly, that sampling objectives are met, and that chain of custody procedures are being followed.
7. Verify that sample extraction and chemical analysis occurs according to documented procedures. Assure that suitable and sufficient analytical quality control samples and reference standards are analyzed.
8. Verify that analytical data calculations are properly generated and the data are correctly associated with the proper samples.
9. Assure that conclusions based on the chemical analysis of the samples are in keeping with the sampling procedures and sample site locations.
10. Document quality assurance activities including: who did it, what was done, when it was done, where was it done, and why was it

done. Document and justify any deviations from documented procedures and policies.

**An Example of  
Procedures to Use When Cleaning Wipe Sampling Equipment**

1. Using clean (or cleaned) disposable equipment is overall probably more cost-effective than cleaning and verifying that cleaned sampling equipment is free from PCBs. The second choice is not cleaning any equipment on or near the sampling site, but to have sufficient recleaned sampling equipment to completely sample a site. The least favorable situation is to clean sampling equipment for reuse at the same sampling site. If cleaning must be done at or near the sampling site, clean the sampling equipment as far from the actual site of cleanup/contaminations as possible.

2. Try to have sufficient clean materials on-site to completely sample a site (plus at least ten percent surplus for unforeseen accidents and blunders) so as not to have to clean any sampling equipment.

3. Use cleaning procedures which have been verified as effective previously. Good cleaning includes:

- Washing with soapy water
- Rinsing thoroughly with water
- Rinsing three times thoroughly with distilled water
- Rinsing with PCB-free organic solvent
- Air drying for non-glass
- Drying in a muffle furnace at 350°C for glass
- Verification sampling and analysis of cleaned equipment
- Protective packaging for shipment to the sampling site

4. The same kind of verification procedures should be used for new equipment as is used for equipment which has been cleaned:

a. Selecting a statistical sample from the equipment. For lots having large numbers of units (such as sample bottles), a 5% or less proportion of the units may be sufficient. For equipment which comes in direct contact with contaminated surfaces (such as templates) a 10% sample may be more appropriate unless historical data have verified that a smaller proportion is sufficient.

b. Rinsing "clean", dry equipment with the same amount of organic solvent as is used in the sampling procedure or more than sufficient solvent to completely cover and rinse off all contact (with the wipe sample, sampler, or the surface) surfaces of equipment. The rinseate is collected and treated as an extract from a sample gauze pad.

c. The presence of detectable levels of PCBs indicate that

contamination is present and that the lot from which the verification sample(s) came must be either recleaned and reverified or disposed of appropriately.

## **II. DESCRIPTION OF DOUBLE WASH/RINSE**

### **Introduction**

The PCB Spill Cleanup Policy requires that low concentration spills of small amounts of PCBs on surfaces are to be removed by a double wash/rinse procedure. The objectives of the double wash/rinse are (1) to recognize the lesser hazard resulting from these small quantity spills and from the cleanup of such spills, and (2) to remove the easily removable PCB material thoroughly and quickly. It is also important not to redistribute PCBs or leave pieces of cleanup materials as a result of the cleanup procedure.

### **General Requirements for All Double Wash/Rinse Surfaces**

For spills where there is still visible PCB-containing liquid present on the surface to be cleaned up, the double wash/rinse procedure first requires a pre-cleaning step. This step includes thoroughly wiping/mopping up the entire surface with absorbent paper or cloth material, such that there are no longer visible signs of the liquid present on the surface.

The double wash/rinse procedure called for in the cleanup of surfaces contaminated by small spills includes the two washing steps and two rinsing steps. The two washing and rinsing steps are slightly different depending on: (a) whether a contaminated surface was relatively clean before the spill, or (b) whether a surface was coated/covered with some sort of absorbent material, such as dust, dirt, grime, or grease.

Minimization of residual PCBs following the double wash/rinse procedure is facilitated by the proper selection and use of cleanup equipment. Scrubbers and the absorbent pads used in the double wash/rinse procedure shall not be dissolved by solvents or cleaners used. Scrubbers and absorbent pads shall not contain greater than 2 parts per million (weight per weight) PCBs. Washing scrubbers and absorbent pads shall not be reused. Rinsing scrubbers and absorbent pads may be reused as washing scrubbers or absorbent pads if necessary, but this is not recommended. All double wash/rinse cleaning/absorbent materials must remain intact (i.e. do not shred, crumble, or leave visible fragments on the surface) after the double wash/rinse operation.

During the double wash/rinse process, all washing and rinsing liquids/solvents must be contained, captured, and properly disposed of in accordance with local, state, and Federal regulations. Following use in the double wash/rinse process, all double wash/rinse equipment and absorbent materials must also be disposed

of in accordance with local state, and Federal regulations.

## Summary of The Double Wash/Rinse Procedure

### General

1. Use disposable cleaning materials which do not
  - dissolve or break apart
  - contain traces of PCBs.
2. Remove any visible PCB liquid before washing/rinsing.
3. Capture and contain washing/rinsing solutions.
4. Properly dispose of cleaning materials and solutions/liquids.

### Specific

1. For surfaces not covered with dirt, dust, grime, grease or other potential absorbent of PCBs:

WASH 1: Scrub with organic solvent and wipe up the solvent.

RINSE 1: Wipe surface with moistened pad, wipe up with dry pad.

WASH 2: Repeat WASH 1.

RINSE 2: Repeat RINSE 1.

2. For surfaces covered with dirt, dust, grime, grease or other potential absorbent of PCBs:

WASH 1: Scrub with detergent and water, dry.

RINSE 1: Rinse with water, wipe with wet adsorbent pad, dry.

WASH 2: Scrub with organic solvent and wipe up the solvent.

RINSE 2: Wipe surface with moistened pad, wipe up with dry pad.



Detailed Requirements for the Double Wash/Rinse

1. Specific requirements for surfaces that do not appear dusty or grimy before a spill, such as glass, automobile surfaces, newly poured concrete, and desk tops:

WASH 1.

If there is no visible liquid or after having removed the visible liquid, cover the entire surface with organic solvent in which PCBs are soluble to at least 5% by weight. Contain and collect any runoff solvent for disposal. Scrub rough surfaces with a scrub brush or disposable scrubbing pad. Add solvent such that the surface is always very wet for one minute per square foot. Wipe smooth surfaces with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Wipe, mop, and/or sorb the solvent onto absorbent material until no visible traces of the solvent remain.

RINSE 1.

Wipe the surface with an absorbent pad soaked with the same organic solvent with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Immediately wipe/sop up the solvent on the surface with a dry absorbent.

WASH 2.

Repeat WASH 1.

RINSE 2.

Repeat RINSE 1.

Detailed Requirements for the Double Wash/Rinse (Continued)

2. Specific requirements for dirty, dusty, grimy, or greasy surfaces or surfaces having surface coverings of some other kind of sorbant materials (where the spill probably largely sorbed onto the materials on the surface):

WASH 1.

If there is no visible liquid or after having removed the visible liquid, cover the entire surface with concentrated or industrial strength detergent or non-ionic surfactant solution. Contain and collect all cleaning solutions for proper disposal. Scrub rough surfaces with a scrub brush or scrubbing pad, adding cleaning solution such that the surface is always very wet, for one minute per square foot. Wipe smooth surfaces with a cleaning solution-soaked disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Mop up or absorb the residual cleaner solution and suds with an absorbent pad until the surface appears dry. This cleaning should remove any residual dirt, dust, grime, or other sorbant materials left on the surface following step one (above).

RINSE 1.

Rinse off the wash solution with one gallon of water per square foot and capture the rinse water. Mop up the wet surface until the surface appears dry.

WASH 2.

Next, cover the entire dry surface with organic solvent in which PCBs are soluble to at least 5% by weight. Scrub rough surfaces with a scrub brush or scrubbing pad adding solvent such that the surface is always very wet for one minute per square foot. Wipe smooth surfaces with a solvent-soaked, disposable absorbent pad for one minute per square foot. Any surface less than one square foot shall also be washed for one minute. Wipe, mop, and/or sorb the solvent onto absorbent material until no visible traces of the solvent remain.

RINSE 2.

Wipe the surface with an absorbent pad soaked with the

same organic solvent as in RINSE 1 (above) and immediately wipe up the solvent on the surface with a dry absorbent.

**APPENDIX C**

**STANDARD OPERATING PROCEDURES**

## **STANDARD OPERATING PROCEDURE**

### **FIELD ACTIVITY RECORDS**

#### **1.0 PURPOSE**

The purpose of this procedure is to describe the methods for use and maintenance of field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized format.

This procedure provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the URS Project Manager and URS Quality Assurance Manager.

#### **2.0 DEFINITIONS**

##### **2.1 Definitions**

Not applicable.

#### **3.0 RESPONSIBILITIES**

Field personnel are responsible for performing the applicable tasks in accordance with this procedure when conducting work related to environmental projects. Daily logs will be kept during field activities by a Field Team Member to provide daily records of significant events, observations and measurements taken in the field.

The URS Project Manager or an approved designee is responsible for checking all work performance and acknowledging that the applicable tasks required by this procedure have been performed. This will be accomplished by reviewing all documents and data produced during work performance.

#### **4.0 PROCEDURE**

##### **4.1 Introduction**

Field logbooks provide a means for recording all data collecting activities performed at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events which occurred while performing field activities and to refresh the memory of field personnel if called upon to give testimony during legal proceedings. As such, all entries will be as factual, detailed and as descriptive as possible so that a particular situation can be reconstructed without reliance on the collector's memory. Field logbooks are not to be used as a sole source of project or sampling information.

#### **4.2 Field Logbook Identification**

Field logbooks shall be bound with preprinted consecutively numbered pages. Logbooks will be permanently assigned to field personnel for the duration of a project, but are to be stored in site project files when not in use. If site activities stop for an extended period of time (i.e., two weeks or more), field logbooks will be stored in the project files. Prior to the commencement of sampling, logbooks will be assigned to field personnel.

The cover of each logbook will contain the following information:

- Person or organization to whom the book is assigned;
- Book number;
- Project number (if different than site number);
- Site name;
- Start date; and
- End date.

#### **4.3 Logbook Entry Procedure**

Daily logs will be kept during field activities by a Field Team Member to provide daily records of significant events, observations, and measurements during field operations. Beginning on the first blank page and extending through as many pages as necessary, the following list provides examples of useful and pertinent information that may be recorded (optional).

- Serial numbers and model numbers for equipment which will be used for the project duration;
- Formulas, constants, and example calculations;
- Useful phone numbers; and
- County, state, and site address.

Entries into the logbook may contain a variety of information. At a minimum, logbook entries must include the following information at the beginning of each day:

- Date;
- Start time;
- Weather;
- All field personnel present and directly involved;
- Level of personal protection equipment being used on the site;
- Signature of the person making the entry; and
- Equipment used and procedures followed.

In addition, information recorded in the field logbook during the day will include (but is not limited to) the following:

- Sample description including sample numbers, time, depth, volume, containers, preservative, and media sampled;
- Information on field QC samples (i.e., duplicates);
- Observations about site and samples (odors, appearance, etc.);
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples;
- Equipment used on site including time and date of calibration;
- Field parameters (pH, specific conductivity, etc.);
- Maps or photographs acquired or taken at the sampling site, including photograph number and description;
- Forms numbers and any information contained therein used during sampling should be referenced; and

All logbook entries will be made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialed and dated by the originator. Entries will be organized into easily understandable tables if possible.

All log book pages will be initialed and dated at the top of the page. Times will be recorded next to each entry.

No pages or spaces will be left blank. If the last entry for a day is not at the end of the page, a diagonal line will be drawn through the remaining space and the line will be initialed and dated.

#### **4.4 Review**

The URS Project Manager or an approved designee will check field logbooks, and daily logs for completeness and accuracy once each week, at a minimum. Any discrepancies in these documents will be noted and returned to the originator for correction. The reviewer will acknowledge that these review comments have been incorporated by signing and dating the applicable reviewed documents.

#### **5.0 REFERENCES**

U.S. Environmental Protection Agency (EPA). 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." September.

\_\_\_\_\_. 1987. "A Compendium of Superfund Field Operations Methods." EPA/540/P-87/001 (OSWER Directive 9355.0-14). December.

#### **6.0 EXHIBITS**

Not applicable.

## **STANDARD OPERATING PROCEDURE**

### **CHAIN-OF-CUSTODY FORMS**

#### **1.0 PURPOSE**

The purpose of this procedure is to describe the proper chain of custody and tracking methods to be followed for environmental projects. This procedure will outline the documentation necessary to trace sample possession and shipment and will provide standardized forms to be used in the field.

This procedure provides guidance for routine field operations. Site-specific deviations from the methods presented herein must be approved by the URS Project Manager.

#### **2.0 DEFINITIONS**

##### **2.1 Definitions**

Not applicable.

#### **3.0 RESPONSIBILITIES**

Field personnel (samplers) are responsible for performing the tasks in accordance with this procedure. These personnel are responsible for the care and custody of the collected samples until the samples are transferred or dispatched properly.

The URS Project Manager or an approved designee is responsible for checking all work performance and approving that the work satisfies the applicable tasks required in this procedure. This will be accomplished by reviewing all documents (Exhibits) and data produced.

The URS Laboratory Manager is responsible for reviewing the Chain-of-Custody Form to ensure that the requested analyses are correctly written and meet the requirements of the Sampling and Analysis Plan.

#### **4.0 PROCEDURE**

##### **4.1 Introduction**

Samples are collected as described in the Sampling and Analysis Plan.

Written documentation of sample custody from the time of sample collection through the generation of data by analysis of that sample is recognized as a vital aspect of an environmental study. Sample custody consists of three parts: sample collection, laboratory analysis, and final evidence files. The chain of custody of the physical sample and its corresponding documentation will be maintained throughout the handling of the sample. All samples will be identified, labeled, logged onto a Chain-of-Custody Form,

and recorded in a sample tracking log as a part of the procedure to ensure the integrity of the resulting data. The record of the physical sample (location and time of sampling) will be joined with the analytical results through accurate accounting of the sample custody. As described below, sample custody applies to both field and laboratory operations.

A sample or evidence file is under custody if it is in:

- The possession of the sampler/analyst;
- The view of the sampler/analyst, after being in the possession of, the sampler/analyst;
- The possession of the sampler/analyst and then placed in a secured location; or
- A designated secure area.

Waterproof ink will be used unless prohibited by weather conditions. For example, a log book notation will explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

#### **4.2 Transfer of Custody and Sample Tracking Procedures**

Samples will be accompanied by a properly completed Chain-of-Custody Form. An example of a Chain-of-Custody Form is shown in Exhibit 4.2-1. The sample numbers, locations, and requested analyses will be listed. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the laboratory, and to/from a secure storage area.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis. Shipping containers will be secured with strapping tape. Custody Seals will be placed on the shipping container for shipment to the laboratory. The preferred procedure is the attachment of a Custody Seal to the front right and back left of the cooler. The Custody Seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the Chain-of-Custody Forms as long as the Chain-of-Custody Forms are sealed inside the sample cooler and the Custody Seals remain intact.

#### **5.0 REVIEW**

The sampler is responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples. The sampler is also responsible for reviewing (or for having a second sampler review) the custody forms for completeness and accuracy before relinquishing custody.

The URS Project Manager or an approved designee must review all field activities to determine whether proper chain of custody procedures were followed during the field work and to decide if additional samples are required. The sampler should notify the URS Project Manager of a breach or irregularity in chain of custody procedures.

## **6.0 REFERENCES**

U.S. Environmental Protection Agency (EPA). 1978. "Policies and Procedures." EPA/330/9-78-00/-R. NEIC.

\_\_\_\_\_. 1987. "A Compendium of Superfund Field Operations Methods." EPA/540/P-87/001 (OSWER Directive 9355.01-14). December.

\_\_\_\_\_. 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." (OSWER Directive 9950.1). September.

## **7.0 EXHIBITS**

Example Chain-of-Custody Form



## STANDARD OPERATING PROCEDURE

### SAMPLE MANAGEMENT

#### 1.0 PURPOSE

The purpose of this procedure is to list acceptable sample containers and describe sample preservation and maximum holding times to be used during all field investigations for low-, medium-, or high-concentration samples of liquid, soil, sediment and sludge matrices.

This procedure provides guidance for routine field operations on environmental projects.

#### 2.0 DEFINITIONS

##### 2.1 Definitions

*Low-Concentration Sample:* In general, the contaminant of highest concentration is present at a level less than 10 parts per million (ppm). Examples include background environmental samples, perimeter, and lagoon samples.

*Medium-Concentration Sample:* In general, the contaminant of highest concentration is present at a level greater than 10 ppm and less than 15 percent by volume (150,000 ppm). Examples include weathered material.

*High-Concentration Sample:* In general, at least one contaminant is present at a level greater than 15 percent by volume. Samples from drums and tanks are assumed to be high concentration unless information indicates otherwise.

#### 3.0 RESPONSIBILITIES

Sampling personnel (samplers) are responsible for performing the applicable tasks outlined in this procedure.

The URS Project Manager or an approved designee is responsible for checking all work performance and approving that the work satisfies the applicable tasks required by this procedure. This will be accomplished by reviewing all documents and data produced during work performance.

#### 4.0 PROCEDURE

The sampling and analysis program for environmental assignments must comply with the analytical procedures that comply with the applicable regulations.

The purpose of sample preservation is to prevent or retard the degradation and modification of chemicals or to retard biological activity in samples during transit and storage. Efforts to preserve the integrity of the samples must be initiated at the time of sampling and continue until analyses are performed. Preservatives must be added to the

sample container at the time of sample collection. The recommended procedure is to take pre-measured volumes of the preservatives in sealed ampules to the field.

Complete and unequivocal preservation of samples, domestic sewage, industrial wastes, or natural waters, is impossible in practice. Regardless of the nature of the sample, complete stability for every constituent is not likely to be achieved. At best, preservation techniques can retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. Degradation of the sample ceases only if it is preserved at a temperature of absolute zero (-273°C). However, freezing of a sample to extend hold times is not permitted. Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low microgram per liter ( $\mu\text{g/l}$ ) range.

Methods of preservation are relatively limited and are intended generally to perform the following:

- Retard biological action;
- Retard hydrolysis of chemical compounds and complexes;
- Reduce volatility of constituents; and
- Reduce absorption effects.

Preservation methods are generally limited to:

- pH control;
- Chemical addition; and
- Refrigeration.

The recommended preservative for various constituents is given in Table 5 of the SAP. Table 5 also provides the estimated volume of sample required for the analysis, the suggested type of container, and the maximum recommended holding times for samples properly preserved.

**When selecting preservation techniques and sample container type, always refer to the guidance provided in the documentation of the analytical methods to be used.**

#### 4.1 Sample Containers

Select sample containers based on the analytical parameters of interest. Use containers made of materials that are nonreactive. Glass and polyethylene containers are the most commonly accepted, and both are used when sampling many constituents. When metals are the analytes of interest, however, polyethylene containers with polypropylene caps are preferred. When organics are the analytes of interest, use amber glass containers with Teflon-lined caps.

## 4.2 Sample Preservation

Perform appropriate chemical preservation in the field for various analytical parameters at the time of sampling. When appropriate, cool samples after collection and during shipment. All samples should be kept out of direct sunlight and stored in the dark (e.g., in a cooler). Regardless of the method of preservation, perform analyses as soon after sampling as is possible.

In some instances, the optimal method for sample preservation may be inappropriate due to the restrictions placed on the transport of certain chemicals by shippers. When shipping restrictions prevent the use of some reagents for sample preservation, use the most appropriate and permissible technique.

### 4.2.1 Preservation Techniques for Metals

Before sample collection, determine the type of data desired (i.e., dissolved, suspended, total or total recoverable). See Table 5 of the SAP for container preference, maximum holding time and sample preservation at the time of collection. Prepare drinking water samples containing suspended material using the total recoverable metal procedure.

- Dissolved

For the determination of dissolved constituents, filter the sample through a 0.45  $\mu\text{m}$  membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus using plain, non-grid marked, membrane filters are recommended to avoid possible contamination.)

Use the first 50-100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled  $\text{HNO}_3$  to a pH of less than 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the sample. If hexavalent chromium is included in the analytical suite, transfer a portion of the filtrate before acidification to a separate container and analyze as soon as possible. Analyses performed on a sample treated as described will be reported as "dissolved" concentrations.

- Suspended

For the determination of suspended metals, filter a representative volume of unpreserved sample through a 0.45  $\mu\text{m}$  membrane filter.

- Total

For the preservation of total metals, the sample is acidified with 1:1 redistilled  $\text{HNO}_3$  to a pH of less than 2 at the time of collection. The sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. (The sample volume required may also vary proportionally with the number of metals to be determined.)

- Total Recoverable

To determine total recoverable metals, acidify the entire sample at the time of collection with 5 ml of concentrated redistilled  $\text{HNO}_3$  per liter of sample.

- Total Metals in Solid Matrices

For the determination of total metals in solid matrices, the samples are maintained at 4°C from collection through analysis.

#### 4.2.3 Preservation Techniques for Nitrogen Testing

- Ammonia

Add 2 ml concentrated H<sub>2</sub>SO<sub>4</sub> per liter of sample and cool to 4°C.

- Kjeldahl, Total

Preserve by adding 2 ml of concentrated H<sub>2</sub>SO<sub>4</sub> per liter and store at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Analyze preserved samples as soon as possible.

- Nitrate Plus Nitrite, Nitrate

Analyze as soon as possible. If analysis can be made within 24 hours, preserve the sample by refrigeration at 4°C. When samples must be stored for more than 24 hours, preserve them with sulfuric acid (2 ml H<sub>2</sub>SO<sub>4</sub> per liter) and refrigeration.

**Caution:** Samples for reduction column must not be preserved with mercuric chloride.

- Nitrite

Analyze samples as soon as possible. They may be stored for 24 to 48 hours at 4°C.

#### 4.2.4 Preservation Techniques for Organics

- Volatiles

Collect the sample in 40 ml glass bottles with Teflon®-lined septa.

A delay between sampling and analysis of greater than 4 hours requires sample preservation. Preserve sample with hydrochloric acid to a pH of less than 2 and maintain at 4°C until analysis.

Be certain that no air bubbles are present in the bottles. This can be accomplished by inverting the filled and capped bottles and verifying that no air bubbles rise to the bottom of the bottle. If bubbles are present, remove the cap and refill the bottle.

- Chemical Oxygen Demand

Collect the samples only in glass bottles with Teflon®-lined caps.

Test biologically active samples as soon as possible.

The collection of a composite sample and/or division of the sample into separate aliquots is not generally possible due to losses on equipment and imperfect mixing. Preserve samples with sulfuric acid to a pH  $<2$  and maintained at 4°C until analysis.

- Oil and Grease; Petroleum Hydrocarbons

Collect a representative sample of 1 liter volume in a glass bottle. Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. The entire sample is consumed by this test; no other analysis may be performed using aliquots of the sample.

A delay between sampling and analysis of more than 4 hours requires sample preservation by adding 5 ml HCl. A delay greater than 48 hours also requires refrigeration for sample preservation.

- Organic Carbon

Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles, such as conventional polyethylene containers, is not permissible.

Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, minimize the lapse of time between collection and analysis. Also, keep samples cool (4°C) and protect them from sunlight and atmospheric oxygen.

In instances where analysis cannot be performed within two hours (2 hours) from the time of sampling, acidify (pH  $\leq 2$ ) the sample with HCl or H<sub>2</sub>SO<sub>4</sub>.

- Phenolics

Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. Keep the sample cool (4°C) and analyze it within 24 hours after collection.

#### 4.3 Maximum Holding Time

Complete and unequivocal preservation of a sample for an extended period of time is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent is not likely to be achieved. Maximum holding times are assigned to each analyte and are designed for quality assurance purposes to minimize degradation effects on the analysis. Therefore, as a rule, it is better to analyze the sample as soon as possible after collection. This is especially true when low contaminant concentrations are expected. Maximum holding times for analyses specified for this project are presented in Table 5 of the SAP.

#### 4.4 Review

The URS Laboratory Manager or an approved designee shall check all sample control documentation to ensure that the samples, transport, and analysis events have met the criteria outlined in this SOP. Any discrepancies shall be noted and returned to the originator for correction. The reviewer will acknowledge that corrections have been incorporated by signing and dating each reviewed document.

## 5.0 REFERENCE

APHA. 1983. "Standard Methods for the Examination of Water and Wastewater." 14th ed.

U.S. Environmental Protection Agency (EPA). 1983. "Methods for the Chemical Analysis of Water and Wastes." EPA-600/4-79-020. March.

\_\_\_\_\_. 1986. "Test Methods for Evaluating Solid Waste." SW-846.

\_\_\_\_\_. 1983. "RCRA Permit Writer's Manual: Groundwater Protection" (40 CFR Part 264, Subpart F), Geotrans Inc., EPA Contract No. 68-01-6464.

\_\_\_\_\_. 1984. Federal Register Part VIII, 40 CFR Part 136, October 26.

\_\_\_\_\_. 1988. "Users Guide to the Contract Laboratory Program." 9240.0-1. December.

USEPA/NWWA. 1981. "Manual of Groundwater Sampling Procedures." NWWA/EPA Series.

## **STANDARD OPERATING PROCEDURE**

### **FIELD ACTIVITY RECORDS**

#### **1.0 PURPOSE**

The purpose of this procedure is to describe the methods for use and maintenance of field logbooks. This procedure outlines methods, lists examples for proper data entry into a field logbook, and provides the standardized format.

This procedure provides guidance for routine field operations on environmental projects. Site-specific deviations from the methods presented herein must be approved by the URS Project Manager and URS Quality Assurance Manager.

#### **2.0 DEFINITIONS**

##### **2.1 Definitions**

Not applicable.

#### **3.0 RESPONSIBILITIES**

Field personnel are responsible for performing the applicable tasks in accordance with this procedure when conducting work related to environmental projects. Daily logs will be kept during field activities by a Field Team Member to provide daily records of significant events, observations and measurements taken in the field.

The URS Project Manager or an approved designee is responsible for checking all work performance and acknowledging that the applicable tasks required by this procedure have been performed. This will be accomplished by reviewing all documents and data produced during work performance.

#### **4.0 PROCEDURE**

##### **4.1 Introduction**

Field logbooks provide a means for recording all data collecting activities performed at a site. Field logbooks are intended to provide sufficient data and observation notes to enable participants to reconstruct events which occurred while performing field activities and to refresh the memory of field personnel if called upon to give testimony during legal proceedings. As such, all entries will be as factual, detailed and as descriptive as possible so that a particular situation can be reconstructed without reliance on the collector's memory. Field logbooks are not to be used as a sole source of project or sampling information.

## **4.2 Field Logbook Identification**

Field logbooks shall be bound with preprinted consecutively numbered pages. Logbooks will be permanently assigned to field personnel for the duration of a project, but are to be stored in site project files when not in use. If site activities stop for an extended period of time (i.e., two weeks or more), field logbooks will be stored in the project files. Prior to the commencement of sampling, logbooks will be assigned to field personnel.

The cover of each logbook will contain the following information:

- Person or organization to whom the book is assigned;
- Book number;
- Project number (if different than site number);
- Site name;
- Start date; and
- End date.

## **4.3 Logbook Entry Procedure**

Daily logs will be kept during field activities by a Field Team Member to provide daily records of significant events, observations, and measurements during field operations. Beginning on the first blank page and extending through as many pages as necessary, the following list provides examples of useful and pertinent information that may be recorded (optional).

- Serial numbers and model numbers for equipment which will be used for the project duration;
- Formulas, constants, and example calculations;
- Useful phone numbers; and
- County, state, and site address.

Entries into the logbook may contain a variety of information. At a minimum, logbook entries must include the following information at the beginning of each day:

- Date;
- Start time;
- Weather;
- All field personnel present and directly involved;
- Level of personal protection equipment being used on the site;
- Signature of the person making the entry; and
- Equipment used and procedures followed.

In addition, information recorded in the field logbook during the day will include (but is not limited to) the following:

- Sample description including sample numbers, time, depth, volume, containers, preservative, and media sampled;
- Information on field QC samples (i.e., duplicates);
- Observations about site and samples (odors, appearance, etc.);
- Information about any activities, extraneous to sampling activities, that may affect the integrity of the samples;
- Equipment used on site including time and date of calibration;
- Field parameters (pH, specific conductivity, etc.);
- Maps or photographs acquired or taken at the sampling site, including photograph number and description;
- Forms numbers and any information contained therein used during sampling should be referenced; and

All logbook entries will be made in indelible black or blue ink. No erasures are permitted. If an incorrect entry is made, the data will be crossed out with a single strike mark and initialed and dated by the originator. Entries will be organized into easily understandable tables if possible.

All log book pages will be initialed and dated at the top of the page. Times will be recorded next to each entry.

No pages or spaces will be left blank. If the last entry for a day is not at the end of the page, a diagonal line will be drawn through the remaining space and the line will be initialed and dated.

#### **4.4 Review**

The URS Project Manager or an approved designee will check field logbooks, and daily logs for completeness and accuracy once each week, at a minimum. Any discrepancies in these documents will be noted and returned to the originator for correction. The reviewer will acknowledge that these review comments have been incorporated by signing and dating the applicable reviewed documents.

#### **5.0 REFERENCES**

U.S. Environmental Protection Agency (EPA). 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." September.

\_\_\_\_\_. 1987. "A Compendium of Superfund Field Operations Methods." EPA/540/P-87/001 (OSWER Directive 9355.0-14). December.

#### **6.0 EXHIBITS**

Not applicable.

## **STANDARD OPERATING PROCEDURE**

### **CHAIN-OF-CUSTODY FORMS**

#### **1.0 PURPOSE**

The purpose of this procedure is to describe the proper chain of custody and tracking methods to be followed for environmental projects. This procedure will outline the documentation necessary to trace sample possession and shipment and will provide standardized forms to be used in the field.

This procedure provides guidance for routine field operations. Site-specific deviations from the methods presented herein must be approved by the URS Project Manager.

#### **2.0 DEFINITIONS**

##### **2.1 Definitions**

Not applicable.

#### **3.0 RESPONSIBILITIES**

Field personnel (samplers) are responsible for performing the tasks in accordance with this procedure. These personnel are responsible for the care and custody of the collected samples until the samples are transferred or dispatched properly.

The URS Project Manager or an approved designee is responsible for checking all work performance and approving that the work satisfies the applicable tasks required in this procedure. This will be accomplished by reviewing all documents (Exhibits) and data produced.

The URS Laboratory Manager is responsible for reviewing the Chain-of-Custody Form to ensure that the requested analyses are correctly written and meet the requirements of the Sampling and Analysis Plan.

#### **4.0 PROCEDURE**

##### **4.1 Introduction**

Samples are collected as described in the Sampling and Analysis Plan.

Written documentation of sample custody from the time of sample collection through the generation of data by analysis of that sample is recognized as a vital aspect of an environmental study. Sample custody consists of three parts: sample collection, laboratory analysis, and final evidence files. The chain of custody of the physical sample and its corresponding documentation will be maintained throughout the handling of the sample. All samples will be identified, labeled, logged onto a Chain-of-Custody Form,

and recorded in a sample tracking log as a part of the procedure to ensure the integrity of the resulting data. The record of the physical sample (location and time of sampling) will be joined with the analytical results through accurate accounting of the sample custody. As described below, sample custody applies to both field and laboratory operations.

A sample or evidence file is under custody if it is in:

- The possession of the sampler/analyst;
- The view of the sampler/analyst, after being in the possession of, the sampler/analyst;
- The possession of the sampler/analyst and then placed in a secured location; or
- A designated secure area.

Waterproof ink will be used unless prohibited by weather conditions. For example, a log book notation will explain that a pencil was used to fill out the sample tag because the ballpoint pen would not function in freezing weather.

#### **4.2 Transfer of Custody and Sample Tracking Procedures**

Samples will be accompanied by a properly completed Chain-of-Custody Form. An example of a Chain-of-Custody Form is shown in Exhibit 4.2-1. The sample numbers, locations, and requested analyses will be listed. When transferring the possession of samples, the individuals relinquishing and receiving will sign, date, and note the time on the record. This record documents transfer of custody of samples from the sampler to another person, to the laboratory, and to/from a secure storage area.

Samples will be properly packaged for shipment and dispatched to the appropriate laboratory for analysis. Shipping containers will be secured with strapping tape. Custody Seals will be placed on the shipping container for shipment to the laboratory. The preferred procedure is the attachment of a Custody Seal to the front right and back left of the cooler. The Custody Seals are covered with clear plastic tape. The cooler is strapped shut with strapping tape in at least two locations.

If the samples are sent by common carrier, a bill of lading will be used. Receipts of bills of lading will be retained as part of the permanent documentation. Commercial carriers are not required to sign off on the Chain-of-Custody Forms as long as the Chain-of-Custody Forms are sealed inside the sample cooler and the Custody Seals remain intact.

#### **5.0 REVIEW**

The sampler is responsible for the care and custody of the samples until they are transferred or properly dispatched. As few people as possible will handle the samples. The sampler is also responsible for reviewing (or for having a second sampler review) the custody forms for completeness and accuracy before relinquishing custody.

The URS Project Manager or an approved designee must review all field activities to determine whether proper chain of custody procedures were followed during the field work and to decide if additional samples are required. The sampler should notify the URS Project Manager of a breach or irregularity in chain of custody procedures.

## **6.0 REFERENCES**

U.S. Environmental Protection Agency (EPA). 1978. "Policies and Procedures." EPA/330/9-78-00/-R. NEIC.

\_\_\_\_\_. 1987. "A Compendium of Superfund Field Operations Methods." EPA/540/P-87/001 (OSWER Directive 9355.01-14). December.

\_\_\_\_\_. 1986. "RCRA Groundwater Monitoring Technical Enforcement Guidance Document." (OSWER Directive 9950.1). September.

## **7.0 EXHIBITS**

Example Chain-of-Custody Form



## STANDARD OPERATING PROCEDURE

### SAMPLE MANAGEMENT

#### 1.0 PURPOSE

The purpose of this procedure is to list acceptable sample containers and describe sample preservation and maximum holding times to be used during all field investigations for low-, medium-, or high-concentration samples of liquid, soil, sediment and sludge matrices.

This procedure provides guidance for routine field operations on environmental projects.

#### 2.0 DEFINITIONS

##### 2.1 Definitions

*Low-Concentration Sample:* In general, the contaminant of highest concentration is present at a level less than 10 parts per million (ppm). Examples include background environmental samples, perimeter, and lagoon samples.

*Medium-Concentration Sample:* In general, the contaminant of highest concentration is present at a level greater than 10 ppm and less than 15 percent by volume (150,000 ppm). Examples include weathered material.

*High-Concentration Sample:* In general, at least one contaminant is present at a level greater than 15 percent by volume. Samples from drums and tanks are assumed to be high concentration unless information indicates otherwise.

#### 3.0 RESPONSIBILITIES

Sampling personnel (samplers) are responsible for performing the applicable tasks outlined in this procedure.

The URS Project Manager or an approved designee is responsible for checking all work performance and approving that the work satisfies the applicable tasks required by this procedure. This will be accomplished by reviewing all documents and data produced during work performance.

#### 4.0 PROCEDURE

The sampling and analysis program for environmental assignments must comply with the analytical procedures that comply with the applicable regulations.

The purpose of sample preservation is to prevent or retard the degradation and modification of chemicals or to retard biological activity in samples during transit and storage. Efforts to preserve the integrity of the samples must be initiated at the time of sampling and continue until analyses are performed. Preservatives must be added to the

sample container at the time of sample collection. The recommended procedure is to take pre-measured volumes of the preservatives in sealed ampules to the field.

Complete and unequivocal preservation of samples, domestic sewage, industrial wastes, or natural waters, is impossible in practice. Regardless of the nature of the sample, complete stability for every constituent is not likely to be achieved. At best, preservation techniques can retard the chemical and biological changes that inevitably continue after the sample is removed from the parent source. Degradation of the sample ceases only if it is preserved at a temperature of absolute zero (-273°C). However, freezing of a sample to extend hold times is not permitted. Therefore, as a general rule, it is best to analyze the samples as soon as possible after collection. This is especially true when the analyte concentration is expected to be in the low microgram per liter ( $\mu\text{g/l}$ ) range.

Methods of preservation are relatively limited and are intended generally to perform the following:

- Retard biological action;
- Retard hydrolysis of chemical compounds and complexes;
- Reduce volatility of constituents; and
- Reduce absorption effects.

Preservation methods are generally limited to:

- pH control;
- Chemical addition; and
- Refrigeration.

The recommended preservative for various constituents is given in Table 5 of the SAP. Table 5 also provides the estimated volume of sample required for the analysis, the suggested type of container, and the maximum recommended holding times for samples properly preserved.

**When selecting preservation techniques and sample container type, always refer to the guidance provided in the documentation of the analytical methods to be used.**

#### 4.1 Sample Containers

Select sample containers based on the analytical parameters of interest. Use containers made of materials that are nonreactive. Glass and polyethylene containers are the most commonly accepted, and both are used when sampling many constituents. When metals are the analytes of interest, however, polyethylene containers with polypropylene caps are preferred. When organics are the analytes of interest, use amber glass containers with Teflon-lined caps.

## 4.2 Sample Preservation

Perform appropriate chemical preservation in the field for various analytical parameters at the time of sampling. When appropriate, cool samples after collection and during shipment. All samples should be kept out of direct sunlight and stored in the dark (e.g., in a cooler). Regardless of the method of preservation, perform analyses as soon after sampling as is possible.

In some instances, the optimal method for sample preservation may be inappropriate due to the restrictions placed on the transport of certain chemicals by shippers. When shipping restrictions prevent the use of some reagents for sample preservation, use the most appropriate and permissible technique.

### 4.2.1 Preservation Techniques for Metals

Before sample collection, determine the type of data desired (i.e., dissolved, suspended, total or total recoverable). See Table 5 of the SAP for container preference, maximum holding time and sample preservation at the time of collection. Prepare drinking water samples containing suspended material using the total recoverable metal procedure.

- Dissolved

For the determination of dissolved constituents, filter the sample through a 0.45  $\mu\text{m}$  membrane filter as soon as practical after collection. (Glass or plastic filtering apparatus using plain, non-grid marked, membrane filters are recommended to avoid possible contamination.)

Use the first 50-100 ml to rinse the filter flask. Discard this portion and collect the required volume of filtrate. Acidify the filtrate with 1:1 redistilled  $\text{HNO}_3$  to a pH of less than 2. Normally, 3 ml of (1:1) acid per liter should be sufficient to preserve the sample. If hexavalent chromium is included in the analytical suite, transfer a portion of the filtrate before acidification to a separate container and analyze as soon as possible. Analyses performed on a sample treated as described will be reported as "dissolved" concentrations.

- Suspended

For the determination of suspended metals, filter a representative volume of unpreserved sample through a 0.45  $\mu\text{m}$  membrane filter.

- Total

For the preservation of total metals, the sample is acidified with 1:1 redistilled  $\text{HNO}_3$  to a pH of less than 2 at the time of collection. The sample is not filtered before processing. Choose a volume of sample appropriate for the expected level of metals. (The sample volume required may also vary proportionally with the number of metals to be determined.)

- Total Recoverable

To determine total recoverable metals, acidify the entire sample at the time of collection with 5 ml of concentrated redistilled  $\text{HNO}_3$  per liter of sample.

- Total Metals in Solid Matrices

For the determination of total metals in solid matrices, the samples are maintained at 4°C from collection through analysis.

#### 4.2.3 Preservation Techniques for Nitrogen Testing

- Ammonia

Add 2 ml concentrated H<sub>2</sub>SO<sub>4</sub> per liter of sample and cool to 4°C.

- Kjeldahl, Total

Preserve by adding 2 ml of concentrated H<sub>2</sub>SO<sub>4</sub> per liter and store at 4°C. Even when preserved in this manner, conversion of organic nitrogen to ammonia may occur. Analyze preserved samples as soon as possible.

- Nitrate Plus Nitrite, Nitrate

Analyze as soon as possible. If analysis can be made within 24 hours, preserve the sample by refrigeration at 4°C. When samples must be stored for more than 24 hours, preserve them with sulfuric acid (2 ml H<sub>2</sub>SO<sub>4</sub> per liter) and refrigeration.

Caution: Samples for reduction column must not be preserved with mercuric chloride.

- Nitrite

Analyze samples as soon as possible. They may be stored for 24 to 48 hours at 4°C.

#### 4.2.4 Preservation Techniques for Organics

- Volatiles

Collect the sample in 40 ml glass bottles with Teflon®-lined septa.

A delay between sampling and analysis of greater than 4 hours requires sample preservation. Preserve sample with hydrochloric acid to a pH of less than 2 and maintain at 4°C until analysis.

Be certain that no air bubbles are present in the bottles. This can be accomplished by inverting the filled and capped bottles and verifying that no air bubbles rise to the bottom of the bottle. If bubbles are present, remove the cap and refill the bottle.

- Chemical Oxygen Demand

Collect the samples only in glass bottles with Teflon®-lined caps.

Test biologically active samples as soon as possible.

The collection of a composite sample and/or division of the sample into separate aliquots is not generally possible due to losses on equipment and imperfect mixing. Preserve samples with sulfuric acid to a pH <2 and maintained at 4°C until analysis.

- Oil and Grease; Petroleum Hydrocarbons

Collect a representative sample of 1 liter volume in a glass bottle. Because losses of grease will occur on sampling equipment, the collection of a composite sample is impractical. The entire sample is consumed by this test; no other analysis may be performed using aliquots of the sample.

A delay between sampling and analysis of more than 4 hours requires sample preservation by adding 5 ml HCl. A delay greater than 48 hours also requires refrigeration for sample preservation.

- Organic Carbon

Sampling and storage of samples in glass bottles is preferable. Sampling and storage in plastic bottles, such as conventional polyethylene containers, is not permissible.

Because of the possibility of oxidation or bacterial decomposition of some components of aqueous samples, minimize the lapse of time between collection and analysis. Also, keep samples cool (4°C) and protect them from sunlight and atmospheric oxygen.

In instances where analysis cannot be performed within two hours (2 hours) from the time of sampling, acidify (pH ≤2) the sample with HCl or H<sub>2</sub>SO<sub>4</sub>.

- Phenolics

Biological degradation is inhibited by the addition of 1 g/l of copper sulfate to the sample and acidification to a pH of less than 4 with phosphoric acid. Keep the sample cool (4°C) and analyze it within 24 hours after collection.

### 4.3 Maximum Holding Time

Complete and unequivocal preservation of a sample for an extended period of time is a practical impossibility. Regardless of the nature of the sample, complete stability for every constituent is not likely to be achieved. Maximum holding times are assigned to each analyte and are designed for quality assurance purposes to minimize degradation effects on the analysis. Therefore, as a rule, it is better to analyze the sample as soon as possible after collection. This is especially true when low contaminant concentrations are expected. Maximum holding times for analyses specified for this project are presented in Table 5 of the SAP.

### 4.4 Review

The URS Laboratory Manager or an approved designee shall check all sample control documentation to ensure that the samples, transport, and analysis events have met the criteria outlined in this SOP. Any discrepancies shall be noted and returned to the originator for correction. The reviewer will acknowledge that corrections have been incorporated by signing and dating each reviewed document.

## 5.0 REFERENCE

- APHA. 1983. "Standard Methods for the Examination of Water and Wastewater." 14th ed.
- U.S. Environmental Protection Agency (EPA). 1983. "Methods for the Chemical Analysis of Water and Wastes." EPA-600/4-79-020. March.
- \_\_\_\_\_. 1986. "Test Methods for Evaluating Solid Waste." SW-846.
- \_\_\_\_\_. 1983. "RCRA Permit Writer's Manual: Groundwater Protection" (40 CFR Part 264, Subpart F), Geotrans Inc., EPA Contract No. 68-01-6464.
- \_\_\_\_\_. 1984. Federal Register Part VIII, 40 CFR Part 136, October 26.
- \_\_\_\_\_. 1988. "Users Guide to the Contract Laboratory Program." 9240.0-1. December.
- USEPA/NWWA. 1981. "Manual of Groundwater Sampling Procedures." NWWA/EPA Series.

# **STANDARD OPERATING PROCEDURE**

## **1.0 COLLECTION OF SOIL SAMPLES**

This SOP describes the protocols for sample collection of surface and subsurface soil samples for laboratory analysis.

### **1.1 EQUIPMENT AND MATERIALS REQUIRED FOR PROCEDURE**

The following equipment should be used during sample collection activities:

- Appropriate personnel protective equipment (PPE) as per HASP
- Decontamination equipment
- Paper towels
- Waterproof and permanent markers or pens
- Sample shipping containers
- Ziploc or similar plastic bags
- Packing material for sample containers
- Field logbook, field sheets, chain-of-custody sheets, sample collection log, air monitoring log, HASP
- Stainless steel bowl, trowel, knife, and spatula
- Plastic sheeting
- Aluminum foil
- Laboratory supplied sample containers and caps OR brass rings, Teflon sheets and end caps

### **1.2 SAMPLE COLLECTION PROCEDURES**

Each sample location will be cleared of vegetation or debris prior to sample collection. Standard utility clearance procedures will be followed prior to collection of subsurface samples. Each sample location will be marked in accordance with the project specific Field Sampling Plan.

#### **1.2.1 Discrete Surface Sample Collection**

Surface samples (at depths between zero and six inches) will be collected using a decontaminated stainless steel trowel OR by driving a brass ring or sleeve into the soil at the

desired sample location. Personnel will use a clean, chemical resistant pair of gloves for each discrete sampling location to avoid cross-contamination and exposure to contaminants. The sample will be placed in a clean, stainless steel bowl.

Following collection of the sample, mix the sample thoroughly with the decontaminated trowel and/or spatula. The goal is to achieve as homogeneous a mixture as possible. Following mixing, use the clean stainless steel spoon or the stainless steel trowel to transfer the sample into the container. The appropriate sample volume will be determined by the analytical method to be performed. Ensure that the filled sample container will provide the required sample volume. Cap the container tightly.

### **1.2.2 Discrete Subsurface Sample Collection**

Subsurface samples will be collected following advancement of a boring by hand augering, a drill rig, or a push-probe rig as determined based on the desired sample depth and site-specific conditions. Following advancement of the boring, the sample will be collected from undisturbed soil in a brass ring/sleeve, or acetate sleeve depending upon the drilling method used and the site-specific Field Sampling Plan.

The ends of brass rings are to be covered with Teflon sheets, capped, and sealed. Each sample is labeled and placed in a Ziploc or equivalent bag, which is sealed.

### **1.2.3 Sample**

Label the sample container(s) using waterproof ink or marker. Each sample should be labeled with the project name or number, Sample ID (which typically includes location and depth information), and date.

## **1.3 FIELD QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES**

Field QA/QC samples which may be collected include trip blanks, reagent blanks, rinsate blanks, and field duplicates or split samples. Field QA/QC samples will be determined on a project specific basis based on project data quality objectives.

## **1.4 REFERENCES**

- ADEQ, 2000; *Arizona Department of Environmental Quality Superfund Program Section Quality Assurance Program Plan*, Arizona Department of Environmental Quality SPS, May 22, 2000.

- U.S. EPA, SW-846, *Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Revision 4*, Chapter 9, Sampling Methods, December 1996

## **STANDARD OPERATING PROCEDURE**

### **INVESTIGATION-DERIVED WASTE**

#### **1.0 PURPOSE**

The purpose of this Standard Operating Procedure (SOP) is to ensure that Investigation-Derived Waste (IDW) generated as the result of environmental investigations is managed:

- In a manner consistent with the federal and state applicable or relevant and appropriate requirements (ARARs) to the extent practical;
- To ensure that the potential of further contamination to the site from IDW is eliminated;
- To ensure the quantity of generated IDW is minimized;
- To provide labeling, tracking, and inventory of IDW; and
- To reduce health and safety concerns by reducing the potential for exposure.

#### **2.0 SCOPE**

This SOP applies to all contractor personnel and subcontractors generating, transporting, and handling IDW during environmental investigations and monitoring programs at the site. This SOP describes the minimum acceptable practices.

#### **3.0 METHOD**

##### **3.1 General**

The IDW generated should be considered part of the site that is being investigated and should be managed with other wastes from the site. The IDW should be managed in a protective manner and should comply with Arizona Department of Environmental Quality (ADEQ) and Resource Conservation Recovery Act (RCRA) requirements.

##### **3.2 Procedures**

###### ***Materials***

The following materials may be used to comply with this SOP:

- Ring-top 55-gallon drums (DOT-17-H);

- 20-yard (or similar capacity) roll-off boxes with tarps to cover;
- Roll-off liners;
- Drum labels;
- Field supplies such as pallets, Visqueen, rope, drum liners, paint pens, tape, and hand tools;
- Sampling and shipping supplies such as bottles, coolers, plastic bags, vermiculite, self-closing plastic bags, labels, tapes, and bubble wrap;
- Health and safety equipment such as personal protective equipment (PPE), first aid supplies from sites and vehicles, and eye wash stations;
- 250-gallon truck tank(s);
- Submersible pump(s);
- Hoses and clamps; and
- Spill kit that contains absorb booms or pads, vermiculite, hand tools, spill-related PPE.

### *Equipment*

The following equipment will be needed to comply with this SOP:

- Boom truck;
- Flat bed truck;
- Soil sampling equipment such as hand auger, trowels, bowls, sieves, drum thieves, disposable bailers, funnels, and bottles;
- Drum forks or clamps;
- Health and safety equipment and monitoring equipment (photoionization detector [PID], flame ionization detector [FID]);
- First aid supplies, eye wash station, splash protection PPE;
- Water polishing system, if applicable (Project-Specific Basewide Sampling Analysis Plan [SAP] should contain the plans, designs, and equipment list for this system);
- Bulk tank(s); and
- Roll-off boxes.

### ***Solid Investigation-Derived Waste***

All waste that is generated, stored, processed, transported or disposed of in the state must be classified according to Arizona Revised Statutes (ARS) Title 49, Chapter 4, Solid Waste Management, and Chapter 5, Hazardous Waste Disposal.

Non-Indigenous Solid IDW such as disposable sampling equipment or tools will be disposed at a licensed landfill. Used PPE such as Tyvek® or Saranac® suits; latex, nitrile, or rubber gloves or booties; and spent respirator filters will be disposed as hazardous, non-hazardous, or radioactive waste in an appropriate designated drum/container/dumpster based on its generation. PPE generated from coming into contact with listed or characteristic hazardous waste will be disposed in a separate drum/container/dumpster designated for hazardous waste and shipped to an approved facility. However, PPE generated during the investigation from non-hazardous waste areas will be placed in a separate container and will be managed as non-hazardous waste.

Grossly contaminated PPE will be put into a ring-top 55-gallon drum lined with plastic bag drum liner. When the drum is full, the liner bag will be sealed, the lid closed, and the drum will be numbered, labeled, and moved to the secured site drum holding area.

Indigenous Solid IDW Any solids generated during an investigation will be collected and placed in appropriate designated drums/containers/dumpsters and manifested off-site for disposal based on its nature and characteristics as either hazardous waste, non-hazardous waste, or a radioactive waste. For solids where nature of contamination is not known, the contractor must first determine whether the waste is hazardous or non-hazardous using either historical information or sampling. Waste such as drill cuttings, and excess soil samples or other solid media that may be generated, will be stored at the sampling location and/or a designated location at the site. As the IDW is generated, it will be placed into clean ring-top 55-gallon drums or lined roll-off boxes. The drums to be used for the collection of wastes will be placed in new or reconditioned containers carrying the Department of Transportation (DOT) United Nations “performance oriented packaging standard” symbol. Containers storing hazardous waste must be marked using a yellow “Hazardous Waste” label with the name, address, city, state, zip code, EPA ID number, EPA waste number, accumulation start date, manifest document number, and DOT proper shipping name.

### *Solid Waste Sampling and Characterization*

Generators who characterize waste using analytical methods to classify their waste must follow all state requirements. Where required by the disposal facilities, additional samples from each drum/container may be required. The solid samples will be collected using either a trier or an auger (SW-846, Chapter 9, Sampling Plans, 1986). For each drum/container, the analytical data associated with the samples collected for characterization of the waste will be evaluated to determine if the IDW is hazardous waste. To determine the characteristic of the IDW, the analytical results will be compared to the Toxicity Characteristic Leaching Procedure (TCLP) regulatory limit (40 CFR 261.24).

The field sampler will accurately record field-screening data on the waste accumulation log. IDW at each site will be containerized, managed, and sampled as a single waste stream. The non-segregated IDW may be bulk containerized with other non-hazardous solid IDW as necessary for efficient management of the waste.

Grossly contaminated non-indigenous IDW will be characterized as special or hazardous waste. The drums will be labeled, manifested, transported, and disposed of at a licensed RCRA hazardous/special waste landfill.

Indigenous solid IDW will be classified after analytical data are available.

If the IDW is found to be either a special waste or a hazardous waste, then the container will be labeled, manifested, transported, and disposed of at a licensed RCRA hazardous/special waste landfill, or a municipal solid waste (MSW) landfill that can accept Special Wastes. The landfill will be certified according to 40 CFR 300.440, also known as the off-site rule.

### *Analytical Methods*

The hazardous waste characteristics are defined in 40 CFR 261 Subpart C. Those characteristics include ignitability, corrosivity, reactivity, and TCLP levels. In the TCLP procedure, samples are extracted by Method 1311, further analyzed for TCLP metals, TCLP volatile and semivolatile organic compounds. All waste streams shipped off-site for disposal will be analyzed for RCRA characteristics in accordance with 40 CFR 261 Subpart C. For purposes of determining accurate waste classification, any commercial laboratory capable of performing SW-846 analytical method, using internally derived quality control limits, are adequate to meet the Data Quality Objectives (DQOs) for off-site disposal. Typically, the laboratory will be associated with the disposal company and will provide relevant information such as bottle sizes, holding times, and sample preparation.

All samples analyzed for IDW characterization will be performed in compliance with the project-specific plans.

### *Liquid Investigation-Derived Waste*

It is expected that during sampling activities at the site, liquids will be generated. The liquid IDW generated at each location, such as groundwater from the purging and development of monitoring wells and wastewaters from the decontamination of equipment will be characterized to determine types of contaminants present and management process required to handle the waste. Liquid IDW may at the Project Manager and Arizona Department of Environmental Quality (ADEQ) direction be combined from several sites if the IDW is determined to be non-hazardous. Historical results may be used to make this determination. Disposal options for purge water include surface discharge or discharge back to the well from which the water was removed (in accordance with the Aquifer Protection General Permit Type A 1.04), or transport and disposal at a permitted treatment facility. The selected disposal option will be project dependent.

### *Liquid Waste Characterization*

Before any data gathering activity occurs, the presence of a water-immiscible phase will be determined visually or using an oil/water interface probe. If a water-immiscible phase is present, then the IDW generated from that location will be pumped into 55-gallon drums. The drums, which will be sealed, numbered, labeled, and recorded on log sheets, will be left on pallets at the location. A composite sample will be taken and analyzed for hazardous waste characteristics. Before sampling, the drums of water with an immiscible layer will be inspected.

If the IDW is found to be a special or hazardous waste as defined in ARS 49-851 to 868, or 40 CFR 261, Subpart D, or ARS Title 49, Chapter 5, then the container will be labeled, manifested, stored in a secured area with temporary berms, until it can be transported for disposal at a licensed RCRA hazardous/special waste disposal facility.

If analytical results from liquid samples demonstrate the IDW is non-hazardous but contains constituents at levels above City of Phoenix guidelines, the wastes must be containerized and disposed at an offsite facility.

If the analytical results from liquid samples show the IDW to be hazardous, the drummed IDW will be managed and disposed of as a hazardous waste.

Empty drums will be decontaminated and if structurally sound, will be reused. If the drum is not structurally sound, the drum will be decontaminated, crushed, and recycled as scrap.

### *Sampling Liquid IDW*

Representative composite samples will be collected and analyzed to characterize the liquid IDW generated at each site and accumulated in bulk storage tanks, drums or other containers using a Coliwas tube. The samples will be labeled, sealed, recorded on the chain of custody, packaged, and shipped to the laboratory for analysis.

### *Analytical Methods*

The hazardous waste characteristics are defined in 40 CFR 261 Subpart C. Those characteristics include: Toxicity, ignitability, corrosivity, reactivity, and TCLP levels by Method 1311, further analyzed for TCLP metals, TCLP volatile and semivolatile organic compounds. For purposes of determining accurate waste classification, any commercial laboratory capable of performing SW-846 analytical method, using internally derived QC control limits, are adequate to meet the DQOs for offsite disposal. Typically, the laboratory will be associated with the disposal company and will provide relevant information such as bottle sizes, holding times, and sample preparation.

### *IDW Management Procedures*

#### *Drum Specifications*

The drums to be used for the collection of wastes will be placed in new or reconditioned containers carrying the DOT UN “performance oriented packaging standard” symbol. Drums should be selected on their ability to hold hazardous, radioactive, and non-hazardous liquids and solids without damage to drum integrity. Special care should be taken in the selection of drums in areas where wastes will be collected and that are known to be reactive, corrosive, or contain organic solvents. The drums will be marked with weather proof indelible ink showing the sampling location (associated with the waste), and an identification number which will represent the location in the storage building where the drum is housed. These markings will be located on the top and side of the drum. Each drum will be stored in a manner so that the drum and attached labels can be inspected without moving the drum or surrounding drums.

#### *Waste Tracking Labels*

Container labels will be placed on the side and top of each container and will include the following information: Waste tracking number; generator; contents; the estimated depth collected (if solid); date the waste was first put in the container; date the container was closed; estimated quantity; and the name, address, and phone number of an emergency Point of Contact

for additional information concerning the containers, and the words “Waste Solids or Waste Liquids”. The drums will comply with DOT regulations outlined in 40 CFR.

### *Transport of Drums and Rolloff Boxes*

All drums will be placed on a flat bed truck or trailer using a forklift or Bobcat and transported to the designated central IDW staging area. Roll-off boxes will be transported on a flatbed trailer to the central IDW staging area when two-thirds full or after the roll-off box is no longer needed. Straps will be used to secure the drums when they are placed on the flat bed and while being transported to the central IDW staging area.

### *Drum Inspections*

The drums will be stored at the sampling location and/or designated location at the site. The drums will rest directly on a gravel/soil substrate with a secondary containment. Any spills will be reported immediately to the Field Manager and the Project Manager. If drum integrity is compromised, it will be immediately corrected. All drums will remain closed at all times except during accumulation or when waste is being sampled or removed.

Drums will be inspected at least monthly by the Field Manager or his designee who will document in the field logbook or on a checklist the condition of each drum. Initiation of any necessary corrective actions will be the responsibility of Field Manager and coordinated with the ADEQ. Any corrective actions will be discussed with the ADEQ and its contractor's project management personnel.

### *Storage Time Limitations*

Potential RCRA hazardous IDW generated at each site(s) will be temporarily stored for no longer than 90 days from beginning date of collection. According to 40 CFR 262.34(a), a generator may accumulate hazardous waste on-site for 90 days or less without a permit or without having interim status provided that the requirements in 40 CFR 262.34(a) are met. The storage unit will conform to 40 CFR 262.34(a), having a secondary containment system lined with 10-mil plastic. All non-hazardous waste will be stored at the central IDW staging area no longer than one year.

## **4.0 REFERENCES**

40 Code of Federal Regulations (CFR) 261, Subpart D.

Arizona Revised Statutes (ARS) Title 49, Chapters 4 and 5.

## **5.0 RECORDS**

The following records will be maintained by the Field Administrator and retained as project documents and included as appropriate in the Waste Management Report for client deliverables.

- Hazardous Waste Weekly Inspection Forms;
- IDW Inventory Sheet;
- Field log books;
- Certificates of Disposal;
- Water treatment logs, sample collection data sheets, and discharge records; and
- Receipts for material being recycled.

**APPENDIX D**  
**QUALITY ASSURANCE**

## D.1 Precision

Precision is the measure of variability between individual sample measurements under prescribed conditions.

For metals analysis, laboratory analytical precision will be assessed through the analysis of laboratory duplicate (D) samples. Precision will be measured as the relative percent difference (RPD) between these replicate measurements. The QA requirement for laboratory precision is an RPD of less than 35% for soil samples if both results are greater than five times the laboratory reporting limit. If either result is less than or equal to five times the reporting limit, the QA requirement will be agreement within three times the reporting limit for soil samples.

Field sampling and analysis precision will be assessed through the analysis of homogenized field duplicate (FD) samples submitted blind to the laboratory. Concentration dependent evaluation criteria will be used to evaluate overall sampling and analysis precision. For analytes where both the sample and field duplicate results are greater than 5 times the higher of the detection limit or the reporting limits, precision will be evaluated by means of RPD. Acceptable agreement will be indicated by an RPD of less than or equal to 50%. For analytes where either result is less than 5 times the greater of the two reporting limits, field duplicate results are evaluated by comparing the absolute difference between the results to a factor of the higher reporting limit. Acceptable agreement will be indicated by an absolute difference of less than or equal to 3 times the higher reporting limit.

RPD will be calculated in accordance with the following formula:

$$RPD(\%) = \frac{|S - D|}{(S + D)/2} \times 100$$

Where        S        =        First Measured Value  
                  D        =        Second Measured Value

## D.2 Accuracy

Accuracy is the degree of agreement of a measurement to an accepted reference or true value. The accuracy of the laboratory performance in conducting analyses will be assessed through analysis of laboratory control samples (LCS); clean matrices spiked with known amounts of all target analytes. The accuracy will be measured as the percent recovery (%R) of a given target

analyte relative to its known concentration. The QA objective for laboratory accuracy on LCS analyses is a %R within the range specified by the manufacturer of the LCS solution.

The accuracy of the laboratory analysis on the site-specific matrix will be assessed through analysis of matrix spike (MS) samples. The accuracy will be measured as the %R of a given target analyte relative to the level spiked into the sample. The QA requirement for laboratory accuracy on MS analyses is a %R within the range of 75% to 125% for soil MS samples.

%R will be calculated in accordance with the following formula for MS and LCS results:

$$\%R = \frac{X}{T} \times 100$$

Where        X        =        Measured Value  
                  T        =        True Value

### **D.3    Completeness**

The analytical completeness QA requirement for work under this SAP will be 80%. If any data are rejected, all parties will be notified and a determination made of whether or not the rejected data are critical in meeting project objectives. If the data are considered critical, additional sampling and analysis may be required.

Analytical completeness will be calculated as the ratio of acceptable analytical results (including those qualified as estimated during data validation) to the total number of analytical results requested from the laboratory (expressed as a percentage). Completeness will be calculated by the following formula:

$$\%Completeness = \frac{\textit{Acceptable Analytical Results}}{\textit{TotalNumberofAnalyticalResults Requested}}$$

### **D.4    Representativeness**

Representativeness is the degree to which data accurately and precisely represent a characteristic of a population, parameter variations at a sampling point, or an environmental condition. Representativeness will be maintained during the sampling effort by completing all sampling in

compliance with the procedures described or specified in this SAP and will be evaluated using statistical procedures presented in Section 5.0.

#### **D.5 Comparability**

Comparability expresses the confidence with which one data set can be compared to another. Comparability can be related to accuracy and precision as these quantities are measures of data reliability. Data are considered comparable if siting considerations, collection techniques, measurement procedures, methods, and reporting are of equivalent quality for the samples within a given sample set. All analyses will be conducted using standard analytical methods and all samples will be collected following standard operating procedures in order to maximize the comparability of data to be collected under this SAP.

#### **D.6 Sensitivity**

The analytical laboratory must be able to demonstrate that method reporting limits for all analytes evaluated in a clean matrix (i.e. method or preparation blanks) are no more than half of the applicable regulatory limit.

**APPENDIX E**

**TEST AMERICA QUALITY ASSURANCE MANUAL**

# Quality Assurance Manual

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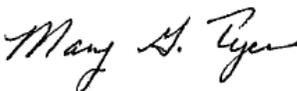
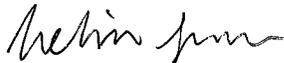
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**Title Page:****Quality Assurance Manual  
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## REFERENCED CORPORATE SOPs AND POLICIES

SOP / Policy Reference	Title
CA-Q-S-001	Solvent and Acid Lot Testing and Approval
CA-Q-S-002	Acceptable Manual Integration Practices
CA-Q-S-004	Method Compliance & Data Authenticity Audits
CA-Q-S-006	Detection Limits
CA-Q-S-008	Management Systems Review
CW-Q-S-001	Corporate Document Control and Archiving
CW-Q-S-002	Writing a Standard Operating Procedure (SOPs)
CW-L-S-002	Internal Investigation of Potential Data Discrepancies and Determination for Data Recall
CA-L-S-002	Subcontracting Procedures
CW-L-P-004	Ethics Policy
CA-L-P-002	Contract Compliance Policy
CW-F-P-002	Authorization Matrix
CW-F-P-004	Procurement and Contracts Policy
CA-C-S-001	Work Sharing Process
CA-T-P-001	Qualified Products List
CW-F-S-007	Controlled Purchases Policy
CW-F-S-018	Vendor Selection
CA-Q-M-002	Corporate Quality Management Plan
CW-E-M-001	Corporate Environmental Health & Safety Manual

## REFERENCED LABORATORY SOPs

SOP Reference	Title
PE-ADM-001	Computer Security
PE-ADM-002	Back-up for Network Data Files
PE-PMD-001	Data Reporting, Validation and Distribution
PE-QAD-001	Control Charts and Statistical Process Control
PE-QAD-002	Pipette Calibration
PE-QAD-003	Sub-sampling
PE-QAD-004	Thermometer Calibration
PE-QAD-006	Logbook Documentation

<b>SOP Reference</b>	<b>Title</b>
PE-QAD-007	Corrective Actions
PE-QAD-008	Personnel Certification and Training
PE-QAD-009	Manual Integration / Data Integrity
PE-QAD-010	Document Control
PE-QAD-012	Receipt Process for General Supplies and Chemicals
PE-QAD-013	Reagent and Standard Preparation, Control and Documentation
PE-QAD-014	Creation and Maintenance of SOPs
PE-QAD-015	Initial Demonstration of Capability
PE-QAD-016	Balance Calibration and Documentation
PE-QAD-017	Record Archiving
PE-QAD-018	Use of Data Qualifiers
PE-QAD-019	Determination of Method Detection Limits
PE-QAD-024	General Data Review
PE-QAD-026	Internal Chain of Custody Procedures
PE-SMP-001	Sample Control
PE-PMD-002	Project Management Communication and Documentation
PE-SMP-004	Field Sampling
PE-SMP-005	Bottle Preparation
PE-SMP-006	Receiving and Waste Management of Foreign Soils
PE-SMP-007	Calibrating Sampling Pumps

## **SECTION 3. INTRODUCTION, SCOPE AND APPLICABILITY**

### **3.1 Introduction and Compliance References**

TestAmerica Phoenix's Quality Assurance Manual (QAM) is a document prepared to define the overall policies, organization objectives and functional responsibilities for achieving TestAmerica's data quality goals. The laboratory maintains a local perspective in its scope of services and client relations and maintains a national perspective in terms of quality.

The QAM has been prepared to assure compliance with The NELAC Institute (TNI) Standard, dated 2009, Volume 1 Modules 2 and 4, AIHA Policies, and ISO/IEC Guide 17025:2005(E). In addition, the policies and procedures outlined in this manual are compliant with TestAmerica's Corporate Quality Management Plan (CQMP) and the various accreditation and certification programs listed in Appendix 3. The CQMP provides a summary of TestAmerica's quality and data integrity system. It contains requirements and general guidelines under which all TestAmerica facilities shall conduct their operations. Please note that the 2003 NELAC standard is based on the 1999 version of 17025.

The QAM has been prepared to be consistent with the requirements of the following documents:

- EPA 600/4-88/039, Methods for the Determination of Organic Compounds in Drinking Water, EPA, Revised July 1991.
- EPA 600/R-95/131, Methods for the Determination of Organic Compounds in Drinking Water, Supplement III, EPA, August 1995.
- EPA 600/4-79-019, Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA, March 1979.
- Test Methods for Evaluating Solid Waste Physical/Chemical Methods (SW846), Third Edition, September 1986, Final Update I, July 1992, Final Update IIA, August 1993, Final Update II, September 1994; Final Update IIB, January 1995; Final Update III, December 1996; Final Update IV, January 2008.
- Federal Register, 40 CFR Parts 136, 141, 172, 173, 178, 179 and 261.
- Statement of Work for Inorganics & Organics Analysis, SOM and ISM, current versions, USEPA Contract Laboratory Program Multi-media, Multi-concentration.
- APHA, Standard Methods for the Examination of Water and Wastewater, 18<sup>th</sup> Edition, 19<sup>th</sup>, 20<sup>th</sup> and on-line Editions.
- U.S. Department of Energy Order 414.1B, Quality Assurance, Approved April 29, 2004.
- Toxic Substances Control Act (TSCA).
- Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA, Second Edition, 1999.
- NIOSH Manual of Analytical Methods, Fourth Edition, 1994.
- U.S. Department of Labor, Occupational Safety & Health Administration, Index of Sampling & Analytical Methods, Revision Date: 21 November 2001.
- AIHA Policies for Laboratory Quality Assurance Programs, 2010 Policy Modules, Effective September 13, 2011.

- Arizona Administrative Code, Department of Health Services, Title 9. Health Services, Chapter 14. Department of Health Services Laboratories, December 31, 2006.
- EPA 815-R-05-004, Manual for the Certification of Laboratories Analyzing Drinking Water, EPA, 5<sup>th</sup> Edition, January 2005.
- New York State Regulations, Title 10 – Department of Health, Chapter 11 – Administrative Rules and Regulations, Part 55 – Approval of Laboratories Performing Environmental Analysis, Revision Date: February 20, 2008.
- Oregon Administrative Rules, Chapter 333, Division 64, October 2000.
- Nevada Administrative Code, Chapter 445A Water Controls – Certification of Laboratories to Analyze Substances in Water; Chapter 445A – Certification of Laboratories to Analyze Drinking Water; November 2008.
- California Environmental Laboratory Improvement Act (Chapter 4 commencing with Section 100825, Part 1, Division 101, of the California Health And Safety Code). ELAP, January 1989.
- California Code of Regulations, Title 22. Social Security, Division 4. Environmental Health, Chapter 19. Certification of Environmental Laboratories; NELAP, January 2000.
- SKC EPA IP-6 Method Update: DETERMINATION OF FORMALDEHYDE AND OTHER ALDEHYDES IN INDOOR AIR; Publication 1661 Rev 1001.
- "Compendium of Methods for the Determination of Pollutants in Indoor Air, "U.S. EPA PB 90-200288, 1990
- American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, 04.09, 1986.

### **3.2 Terms and Definitions**

A Quality Assurance Program is a company-wide system designed to ensure that data produced by the laboratory conforms to the standards set by state and/or federal regulations. The program functions at the management level through company goals and management policies, and at the analytical level through Standard Operating Procedures (SOPs) and quality control. The TestAmerica program is designed to minimize systematic error, encourage constructive, documented problem solving, and provide a framework for continuous improvement within the organization.

Refer to Appendix 2 for the Glossary/Acronyms.

### **3.3 Scope / Fields of Testing**

The laboratory analyzes a broad range of environmental and industrial samples every month. Sample matrices vary among air, drinking water, effluent water, groundwater, hazardous waste, sludge, soils and air for industrial hygiene on varying types of media. 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 processes, reviewing results, servicing clients and tracking samples through the laboratory. The technical and service requirements of all analytical requests are thoroughly evaluated before commitments are made to accept the work. Measurements are made using published reference methods or methods developed and validated by the laboratory.

The methods covered by this manual include the most frequently requested methodologies needed to provide analytical services in the United States and its territories. The specific list of test methods used by the laboratory can be found in Appendix 4. The approach of this manual is to define the minimum level of quality assurance and quality control necessary to meet these requirements. All methods performed by the laboratory shall meet these criteria as appropriate. In some instances, quality assurance project plans (QAPPs), project specific data quality objectives (DQOs) or local regulations may require criteria other than those contained in this manual. In these cases, the laboratory will abide by the requested criteria following review and acceptance of the requirements by the Laboratory Director, Account Executive, Business Development Manager, Client Services Manager and/or the Quality Assurance (QA) Manager. In some cases, QAPPs and DQOs may specify less stringent requirements. The Laboratory Director and/or Industrial Hygiene Program Manager and the QA Manager must determine if it is in the lab's best interest to follow the less stringent requirements.

### **3.4 Management of the Manual**

#### **3.4.1 Review Process**

The template on which this manual is based is reviewed annually by Corporate Quality Management Personnel to assure that it remains in compliance with Section 3.1. This manual is reviewed every two years by senior laboratory management to assure that it reflects current practices and meets the requirements of the laboratory's clients and regulators as well as the CQMP. Occasionally, the manual may need changes in order to meet new or changing regulations and operations. The QA Manager will review the changes in the normal course of business and incorporate changes into revised sections of the document. All updates will be reviewed by the senior laboratory management staff. The laboratory updates and approves such changes according to our Document Control procedures (refer to SOP PE-QAD-010 Document Control).

## **SECTION 4. MANAGEMENT REQUIREMENTS**

### **4.1 Overview**

TestAmerica Phoenix is a local operating unit of TestAmerica Laboratories, Inc. The organizational structure, responsibilities and authorities of the corporate staff of TestAmerica Laboratories, Inc. are presented in the CQMP. The laboratory has day-to-day independent operational authority overseen by corporate officers (e.g., President, Chief Executive Officer, Corporate Quality, etc.). The laboratory operational and support staff work under the direction of the Laboratory Director. The organizational structure for both Corporate & TestAmerica Phoenix is presented in Figure 4-1.

### **4.2 Roles and Responsibilities**

In order for the Quality Assurance Program to function properly, all members of the staff must clearly understand and meet their individual responsibilities as they relate to the quality program. The following descriptions briefly define each role in its relationship to the Quality Assurance Program.

#### **4.2.1 Additional Requirements for Laboratories**

The responsibility for quality resides with every employee of the laboratory. All employees have access to the QAM, are trained to this manual, and are responsible for upholding the standards therein. Each person carries out his/her daily tasks in a manner consistent with the goals and in accordance with the procedures in this manual and the laboratory's SOPs. Role descriptions for Corporate personnel are defined in the Corporate Quality Management Plan (CQMP CA-Q-M-002). This manual is specific to the operations of TestAmerica's Phoenix laboratory.

#### **4.2.2 Laboratory Director**

TestAmerica Phoenix's Laboratory Director is responsible for the overall quality, safety, financial, technical, human resource and service performance of the whole laboratory and reports to their respective GM. The Laboratory Director provides the resources necessary to implement and maintain an effective and comprehensive Quality Assurance and Data Integrity Program. The Laboratory Director who is absent for a period of time exceeding 15 consecutive calendar days shall designate another full time staff member who meets the minimum qualifications of the Laboratory Manager to temporarily perform the Laboratory Director function. Also, if this absence exceeds 65 consecutive calendar days, the primary TNI accrediting authority must be notified in writing.

Specific responsibilities include, but are not limited to:

- Captains the management team, consisting of the QA Manager, the Industrial Hygiene Program Manager, the Business Development Manager, and the Department Manager(s) as direct reports.
- Ensures that all staff has the appropriate education and training to properly carry out the duties assigned to them and ensures that this training has been documented.
- Ensures that personnel are free from any commercial, financial and other undue pressures which might adversely affect the quality of their work.
- Ensures TestAmerica's human resource policies are adhered to and maintained.
- Ensures that sufficient numbers of qualified personnel are employed to supervise and perform the work of the laboratory.
- Ensures that appropriate corrective actions are taken to address analyses identified as requiring such actions by internal and external performance or procedural audits. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs may be temporarily suspended by the Laboratory Director.
- Reviews and approves all SOPs prior to their implementation and ensures all approved SOPs are implemented and adhered to.
- Pursues and maintains appropriate laboratory certification and contract approvals. Supports ISO 17025 requirements.
- Ensures client specific reporting and quality control requirements are met.
- Evaluates the level of internal/external non-conformances for all departments.
- Continuously evaluates production capacity and improves capacity utilization.

- Continuously evaluates turnaround time and addresses any problems that may hinder meeting the required and committed turnaround time from the various departments.
- Develops and improves the training of all analysts in cooperation with the Laboratory Director, the Department Manager(s) and QA Manager and in compliance with regulatory requirements.
- Works to ensure that scheduled instrument maintenance is completed.
- Is responsible for efficient utilization of supplies.
- Constantly monitors and modifies the processing of samples through the departments.
- Fully supports the quality system and, if called upon in the absence of the QA Manager, serves as his/her substitute in the interim.

#### **4.2.3 Quality Assurance (QA) Manager or Designee**

The QA Manager reports directly to the Laboratory Director and has access to Corporate QA for advice and resources. This position is able to evaluate data objectively and perform assessments without outside (e.g., managerial) influence. Corporate QA may be used as a resource in dealing with regulatory requirements, certifications and other quality assurance related items. The QA Manager directs the activities of the QA officers to accomplish specific responsibilities, which include, but are not limited to:

- Serves as the focal point for QA/QC in the laboratory.
- Has functions independent from laboratory operations for which he/she has quality assurance oversight.
- Maintains and updates the QAM.
- Monitors and evaluates laboratory certifications
- Schedules proficiency testing samples.
- Monitors and communicates regulatory changes that may affect the laboratory to management.
- Trains and advises the laboratory staff on quality assurance/quality control procedures that are pertinent to their daily activities.
- Evaluates the thoroughness and effectiveness of training.
- Maintains records of all ethics-related training, including the type and proof of attendance.
- Arranges for or conducts internal audits on quality systems and the technical operation.
- Maintains, improves, and evaluates the corrective action database and the corrective and preventive action systems.
- Notifies laboratory management of deficiencies in the quality system and ensuring corrective action is taken. Procedures that do not meet the standards set forth in the QAM or laboratory SOPs shall be investigated following procedures outlined in Section 12 and if deemed necessary may be temporarily suspended during the investigation.
- Objectively monitors standards of performance in quality control and quality assurance without outside (e.g., managerial) influence.

- Coordinates of document control of SOPs, MDLs, control limits, and miscellaneous forms and information.
- Reviews a percentage of all final data reports for internal consistency. Review of Chain of Custody (COC), correspondence with the analytical request, batch QC status, completeness of any corrective action statements, 5% of calculations, format, holding time, sensibility and completeness of the project file contents.
- Reviews external audit reports and data validation requests.
- Follows up with audits to ensure client QAPP requirements are met.
- Establishes reporting schedule and preparation of various quality reports for the Laboratory Director, clients and/or Corporate QA.
- Develops suggestions and recommendations to improve quality systems.
- Researches current state and federal requirements and guidelines.
- Captains the QA team to enable communication and to distribute duties and responsibilities.
- Ensures monitoring standards of performance to ensure that systems are in place to produce the level of quality as defined in this document.
- The QA Manager has responsibility and authority to ensure the continuous implementation of the quality system based on ISO 17025.
- Has documented training and/or experience in QA/QC procedures and the laboratory's Quality System.
- Has a general knowledge of the analytical test methods for which data audit/review is performed (and/or having the means of getting this information when needed).

Qualifications: The Quality Manager of the laboratory shall possess a bachelor's degree in an applicable basic or applied science and have at least one year of nonacademic analytical or quality control experience appropriate to the types of analyses performed by the laboratory; or quality control experience appropriate to the types of analyses performed by the laboratory; or in lieu of a bachelor's degree, four years of nonacademic analytical or quality control experience. The Quality Manager shall have documented training in statistics or laboratory quality assurance/quality control. The Quality Manager may be a part-time employee or consultant.

NOTE: Appropriate documentation of training in statistics or laboratory quality assurance/quality control shall include at least one of the following: 1) College level course in statistics; 2) Continuing education in laboratory quality assurance/quality control (e.g., AIHA-LAP, LLC or equivalent course); or 3) Relevant experience – documented examples of the level of quality assurance/quality control used in applicable work experience.

#### **4.2.4 Industrial Hygiene Program/Technical Manager**

The Industrial Hygiene Program/Technical Manager reports directly to the Laboratory Director and shall possess the qualifications and assume the responsibilities listed below in addition to the responsibilities listed under the department/program manager title.

- The laboratory shall provide day to day supervision of its technical operations by designating at least one Technical Manager (TM) per program.

- The Industrial Hygiene Program/Technical Manager shall be an employee of the laboratory.
- The Industrial Hygiene Program/Technical Manager shall be present on site at least 20 hours per week or 50 percent of the laboratory operating hours (whichever is less) to address technical issues for laboratory staff and customers.
- The Industrial Hygiene Program/Technical Manager shall authorize and document that all analyses for which the laboratory is accredited are completed by personnel with appropriate education and/or technical background in the Industrial Hygiene department. The Laboratory Director shall have the responsibility to ensure that personnel in other departments, performing industrial hygiene analyses, have appropriate education and/or technical background.
- The Industrial Hygiene Program/Technical Manager shall ensure that adequate supervision is provided for all laboratory technical personnel.
- The Industrial Hygiene Program/Technical Manager or their designee shall function as the approved signatory. The IH Program/Technical Manager/Laboratory Director/Customer Service Manager shall designate those individuals that may function as approved signatories using the Demonstration of Capability form for IH, PX-QAD-005.
- The Industrial Hygiene Program/Technical Manager/Laboratory Director/Customer Service Manager shall designate those individuals that may direct projects from setup through data interpretation and reporting.
- The Industrial Hygiene Program/Technical Manager shall in conjunction with the QA Department Manager ensure on-going proficiency for analysts and technicians that perform analyses that fall under the Industrial Hygiene Program: Every six months a chemist/tech must demonstrate ongoing proficiency. This can be accomplished through the analysis of PAT samples, at least 2 pairs of LCS/LCSD during the six month period, or by repeating the IDC as described in this SOP studies (every six months).
- The Industrial Hygiene Program/Technical Manager shall in conjunction with the QA Department Manager ensure initial/annual reporting level verification spikes are completed as appropriate for each analyte by analysts and technicians that perform analyses that fall under the Industrial Hygiene Program.
- The Industrial Hygiene Program/Technical Manager shall ensure method validation/desorption efficiency studies are performed as appropriate by for analysts and technicians that perform analyses that fall under the Industrial Hygiene Program.
- The Industrial Hygiene Program/Technical Manager shall research and development of new analytical procedures and improvements to current procedures.
- The Industrial Hygiene Program/Technical Manager or their designee, during an absence, shall perform secondary review of all data produced for analyses that fall under the Industrial Hygiene Program.
- The Industrial Hygiene Program/Technical Manager shall possess the following authority:

Stop work on analytical methods that fall under the Industrial Hygiene Program.

Hold or stop issuance of reports that fall under the Industrial Hygiene Program.

Any changes in laboratory ownership, location (except for mobile and field operations laboratories), management, quality control personnel, or any other change that significantly affects the laboratory's capability, scope of accreditation, or ability to meet the policy requirements, shall be reported in writing to AIHA-LAP, LLC within twenty (20) business days of the change. Any absence of personnel for a period in excess of twenty (20) consecutive working days, that impacts the laboratory's ability to perform its scope of testing, shall be reported to AIHA-LAP, LLC within twenty (20) business days. This notification requirement shall be in effect if the Technical Manager, the Quality Manager, or an analyst who is the only staff member that performs a given test, are absent for reasons of extended family leave, illness, temporary disability, etc.

Qualifications of the Industrial Hygiene Program/Technical Manager: Minimum of three (3) years relevant nonacademic analytical experience. A minimum of two (2) years experience shall be in industrial hygiene analyses within the scope of accreditation. The remaining one (1) year may be from other laboratory analytical procedures. Relevant academic experience may be substituted for the remaining one (1) year work experience. A relevant post-graduate degree (MS or Ph.D.) shall also be considered equivalent to one (1) year of work experience. Academic experience and post-graduate degrees may not be substituted for the two (2) years industrial hygiene experience. (Environmental, forensic, or similar microanalytical experience shall be reviewed to determine if the specific experience is a reasonable substitute.) The Industrial Hygiene Program/Technical Manager shall possess a bachelor's degree in an applicable physical or biological science.

#### 4.2.5 Technical Manager or Designee

The Technical Manager(s) report(s) directly to the Laboratory Director. He/she is accountable for all analyses and analysts under their experienced supervision **and for compliance with the ISO 17025 Standard**. The Department Manager acts as a designee for the Technical Director(s). The scope of responsibility ranges from the new-hire process and existing technology through the ongoing training and development programs for existing analysts and new instrumentation. Specific responsibilities include, but are not limited to:

- Exercises day-to-day supervision of laboratory operations for the appropriate field of accreditation and reporting of results. Coordinating, writing, reviewing preparation of all test methods, i. e., SOPs, with regard to quality, integrity, regulatory and optimum and efficient production techniques, and subsequent analyst training and interpretation of the SOPs for implementation and unusual project samples. He/she insures that the SOPs are properly managed and adhered to at the bench. He/she develops standard costing of SOPs to include supplies, labor, overhead, and capacity (design vs. demonstrated versus first-run yield) utilization.
- Reviews and approves, with input from the QA Manager, proposals from marketing, in accordance with an established procedure for the review of requests and contracts. This procedure addresses the adequate definition of methods to be used for analysis and any limitations, the laboratory's capability and resources, the client's expectations. Differences

are resolved before the contract is signed and work begins. A system documenting any significant changes is maintained, as well as pertinent discussions with the client regarding their requirements or the results of the analyses during the performance of the contract. All work subcontracted by the laboratory must be approved by the client. Any deviations from the contract must be disclosed to the client. Once the work has begun, any amendments to the contract must be discussed with the client and so documented.

- Monitors the validity of the analyses performed and data generated in the laboratory. This activity begins with reviewing and supporting all new business contracts, insuring data quality, analyzing internal and external non-conformances to identify root cause issues and implementing the resulting corrective and preventive actions, facilitating the data review process (training, development, and accountability at the bench), and providing technical and troubleshooting expertise on routine and unusual or complex problems.
- Provides training and development programs to applicable laboratory staff as new hires and, subsequently, on a scheduled basis. Training includes instruction on calculations, instrumentation management to include troubleshooting and preventive maintenance.
- Enhances efficiency and improves quality through technical advances and improved LIMS utilization. Capital forecasting and instrument life cycle planning for second generation methods and instruments as well as asset inventory management.
- Coordinates sample management from “cradle to grave,” insuring that no time is lost in locating samples.
- Schedules all QA/QC-related requirements for compliance, e.g., MDLs, etc..
- Captains department personnel to communicate quality, technical, personnel, and instrumental issues for a consistent team approach.
- Coordinates audit responses with the QA Manager.

#### **4.2.6 Environmental Health and Safety Coordinator**

The Environmental Health and Safety Coordinator reports directly to the Laboratory Director and has a dotted line reporting responsibility to the Corporate Environmental Health and Safety Officer. The Environmental Health and Safety Coordinator may also act as the Hazardous Waste Manager or delegate the duties to an authorized and properly trained employee.

- Ensures compliance with air permits.
- Conducts ongoing, required safety training and conduct new employee safety orientation.
- Assists in developing and maintaining the Facility Addendum to the Corporate Employee Health and Safety Manual.
- The Environmental Health and Safety and Hazardous Waste Coordinators shall be tasked with reviewing and updating annually the Hazardous Waste Contingency Plan in the Facility Addendum to the Corporate Environmental Health & Safety Manual
- Administers dispersal of all Material Safety Data Sheet (MSDS) information.
- Performs regular chemical hygiene and housekeeping instruction.
- Gives instruction on proper labeling and practice.
- Serves as chairman of the laboratory safety committee.

- Provides and trains personnel on protective equipment.
- Oversees the inspection and maintenance of general safety equipment – fire extinguishers, safety showers, eyewash stations, etc. and ensure prompt repairs as needed.
- Supervises and schedules fire drills and emergency evacuation drills.
- The Environmental Health and Safety and Hazardous Waste Coordinators shall be tasked to determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- Conducts exposure monitoring assessments when determined necessary.
- Determines when a complaint of possible over-exposure is “reasonable” and should be referred for medical consultation.
- Assists in the internal and external coordination of the medical consultation/monitoring program conducted by TestAmerica’s medical consultants.

#### **4.2.7 Hazardous Waste Coordinator**

The Hazardous Waste Coordinator reports directly to the Laboratory Director. The duties consist of:

- Stays current with the hazardous waste regulations.
- Continues training on hazardous waste issues.
- The Hazardous Waste and Environmental Health and Safety Coordinators shall be tasked with reviewing and updating annually the Hazardous Waste Contingency Plan in the Facility Addendum to the Corporate Environmental Health & Safety Manual.
- Contacts the hazardous waste subcontractors for review of procedures and opportunities for minimization of waste.
- The Hazardous Waste and Environmental Health and Safety Coordinators shall be tasked to determine what initial and subsequent exposure monitoring, if necessary to determine potential employee exposure to chemicals used in the laboratory.
- Ensures proper collection and disposal of all hazardous waste.

#### **4.2.8 Industrial Hygiene Laboratory Analysts/Technicians**

The industrial hygiene program distinguishes two titles for those conducting analytical procedures within the laboratory. An analyst is one who has a bachelor’s degree in chemistry or a related science. A technician is one who does not have a degree in chemistry or a related science.

Analysts and Technicians that perform analyses which fall under the Industrial Hygiene Program shall report directly to the Industrial Hygiene Program/Technical Manager information regarding any analysis that fall under the Industrial Hygiene Program. Analysts and technicians may in addition to the IH Program/Technical Manager report to the designated department manager regarding other non-industrial Hygiene analyses and personnel issues.

Analysts and Technicians that perform analyses which fall under the Industrial Hygiene Program shall possess the qualifications and assume the responsibilities listed below in addition to the responsibilities listed under the Laboratory Analyst title.

This position is responsible for a variety of routine analyses or preparation procedures to determine and evaluate chemical and physical properties. Responsible for interpretation, organization, coordination and completion of routine and/or complex assignments as well as preparation of sampling equipment and materials.

- Successful training (in-house courses are acceptable) in specific methodologies used in the laboratory shall be documented. In house training to be provided on sample preparation and instrument analysis prior to performing independent analysis of laboratory samples. All analysts and technicians shall have a minimum of twenty (20) business days of hands-on experience conducting analyses in an industrial hygiene laboratory before initiation of independent work on customer samples. The criteria and training requirements for laboratory personnel shall be clearly defined, documented and maintained on file in the Quality Assurance office.
- Training Program content, duration, qualifications of the trainer, and objective evidence that the analyst/technician has successfully analyzed unknown reference samples of the matrices/analytes of concern within specified criteria. The dates of authorization to perform specific tasks shall be recorded on the DOC form, PX-QAD-005 and a copy be placed on file in the Quality Assurance office.
- Analysts and Technicians shall have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples. Their performance must be documented.
- Analysts and Technicians shall be responsible for complying with all quality assurance and quality control requirements pertaining to their technical functions.
- Analysts and Technicians shall be responsible to perform on-going proficiency for analyses that fall under the Industrial Hygiene Program: Every six months a chemist/tech must demonstrate ongoing proficiency. This can be accomplished through the analysis of PAT samples, at least 2 pairs of LCS/LCSD during the six month period, or by repeating the IDC as described in the SOP studies (every six months).
- Analysts and Technicians shall be responsible to ensure initial and/or annual reporting level verification spikes are completed as appropriate for each analyte for each method as appropriate, that fall under the Industrial Hygiene Program.
- Analysts and Technicians shall ensure method validation/desorption efficiency studies are performed as appropriate by for analysts and technicians that perform analyses that fall under the Industrial Hygiene Program.
- Analysts and Technicians may assist in research and development of new analytical procedures and improvements to current procedures.
- Analysts and Technicians shall perform preparation and analyses on a variety of samples according to the associated SOP.

- Analysts and Technicians shall train new analysts and technicians in proper use of equipment, maintenance, setup and procedures, as appropriate.
- Analysts and Technicians shall operate, maintain, and trouble shoot as applicable various analytical instrumentation including but not limited to GC-MS, GC, ICP, ICP-MS, cold vapor AA, IC, HPLC UV/VIS, etc.
- Analysts and Technicians shall be responsible to prepare data, perform routine calculations, prepare graphs, tables, and control charts, maintain appropriate organization and cleanliness in lab areas and keep inventory of supplies.

#### **4.2.9 Laboratory Analysts**

Laboratory analysts are responsible for conducting analysis and performing all tasks assigned to them by the Department Manager or designee. The responsibilities of the analysts are listed below:

- Performs analyses by adhering to analytical and quality control protocols prescribed by current SOPs, this QA Manual, and project-specific plans honestly, accurately, timely, safely, and in the most cost-effective manner.
- Documents standard and sample preparation, instrument calibration and maintenance, data calculations, sample matrix effects, and any observed non-conformance on logbooks, benchsheets, lab notebooks and/or the Non-Conformance Database.
- Reports all non-conformance situations, instrument problems, matrix problems and QC failures, which might affect the reliability of the data, to their Department or Program Manager as applicable, and/or the QA Manager or member of QA staff.
- Performs 100% review of the data generated prior to entering and submitting for secondary level review.
- Suggests method improvements to their Department/Program Manager, and the QA Manager. These improvements, if approved, will be incorporated. Ideas for the optimum performance of their assigned area, for example, through the proper cleaning and maintenance of the assigned instruments and equipment, are encouraged.
- Works cohesively as a team in their department to achieve the goals of accurate results, optimum turnaround time, cost effectiveness, cleanliness, complete documentation, and personal knowledge of environmental analysis.
- Lead analyst additional responsibilities - In addition to the responsibilities listed above supports the Technical/Department Manager by monitoring sample throughput, supports adherence to QA and safety protocols, and helps with the daily functions of the department.

#### **4.2.10 LIMS Specialist**

The LIMS Specialist is the individual responsible for the operation, validation, and implementation of the laboratory information management system. LIMS consist of the computer and software used to identify, schedule, prioritize, perform calculations, generate reports, store results, and perform any other computerized function necessary to control the flow

of samples through the laboratory. This person should have a bachelor's degree and/or appropriate laboratory and/or computer skills and education.

#### **4.2.11 Client Services Manager (CSM)**

The Client Services Manager reports to the Laboratory Director and serves as the interface between the laboratory's technical departments and the laboratory's clients. The staff consists of the Project Management team. With the overall goal of total client satisfaction, the functions of this position are outlined below:

- Technical training and growth of the Project Management team.
- Technical liaison for the Project Management team.
- Human resource management of the Project Management team.

The Project Management Team and CSM are:

- Ensures that clients receive the proper sampling supplies.
- Responds to client inquiries concerning sample status.
- Assists clients regarding the resolution of problems concerning COC.
- Ensures that client specifications, when known, are met by communicating project and quality assurance requirements to the laboratory.
- Notifies the Department Managers of incoming projects and sample delivery schedules.
- Accountable to clients for meeting agreed-upon due dates by communicating with the laboratory and relaying pertinent information to the client
- Discusses with client any project-related problems, resolving service issues, and coordinating technical details with the laboratory staff.
- Familiarizes staff with specific quotes, sample log-in review, and final report completeness.
- Monitors the status of all data package projects in-house to ensure timely and accurate delivery of reports.
- Informs clients of data package-related problems and resolve service issues.
- Coordinates requests for sample containers and other services (data packages).

#### **4.2.12 Sample Receiving**

The Sample Receiving Department Manager oversees the Sample Receiving Department. He/She, or designee is responsible for ensuring the timely and correct shipment of sample containers, including proper preservatives and instructions, to clients. He/She maintains accurate records of sample container shipments. The responsibilities are outlined below:

- Directs the logging of incoming samples into the LIMS.
- Ensures the verification of data entry from login.
- Supervises the organized storage and appropriate climate control of samples.

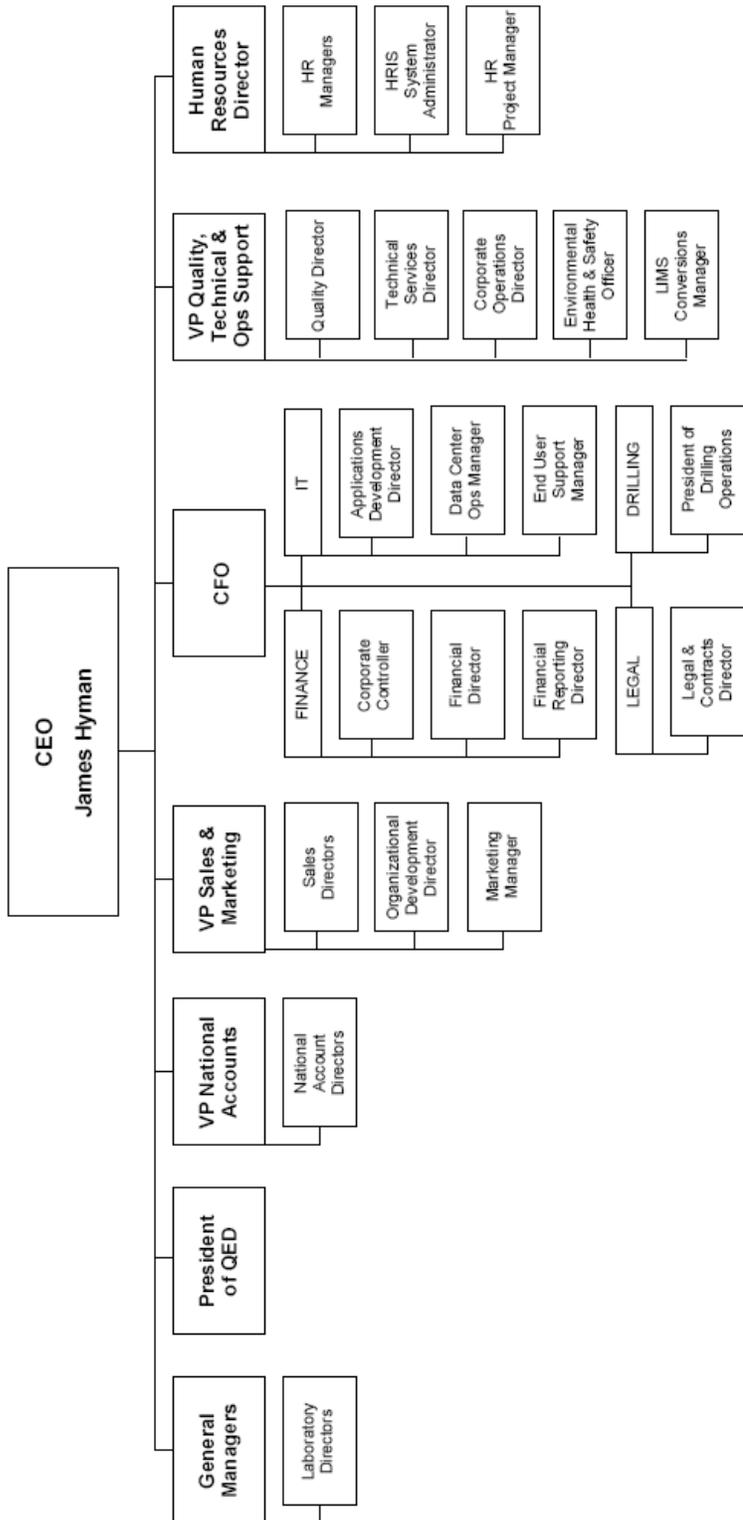
- Supervises the disposal of samples in accordance with the Waste Disposal SOP, the corporate Environmental Health and Safety Manual, the Hazardous Waste Contingency Plan in the facility addendum to the corporate safety manual, and the U. S. Department of Agriculture requirements.

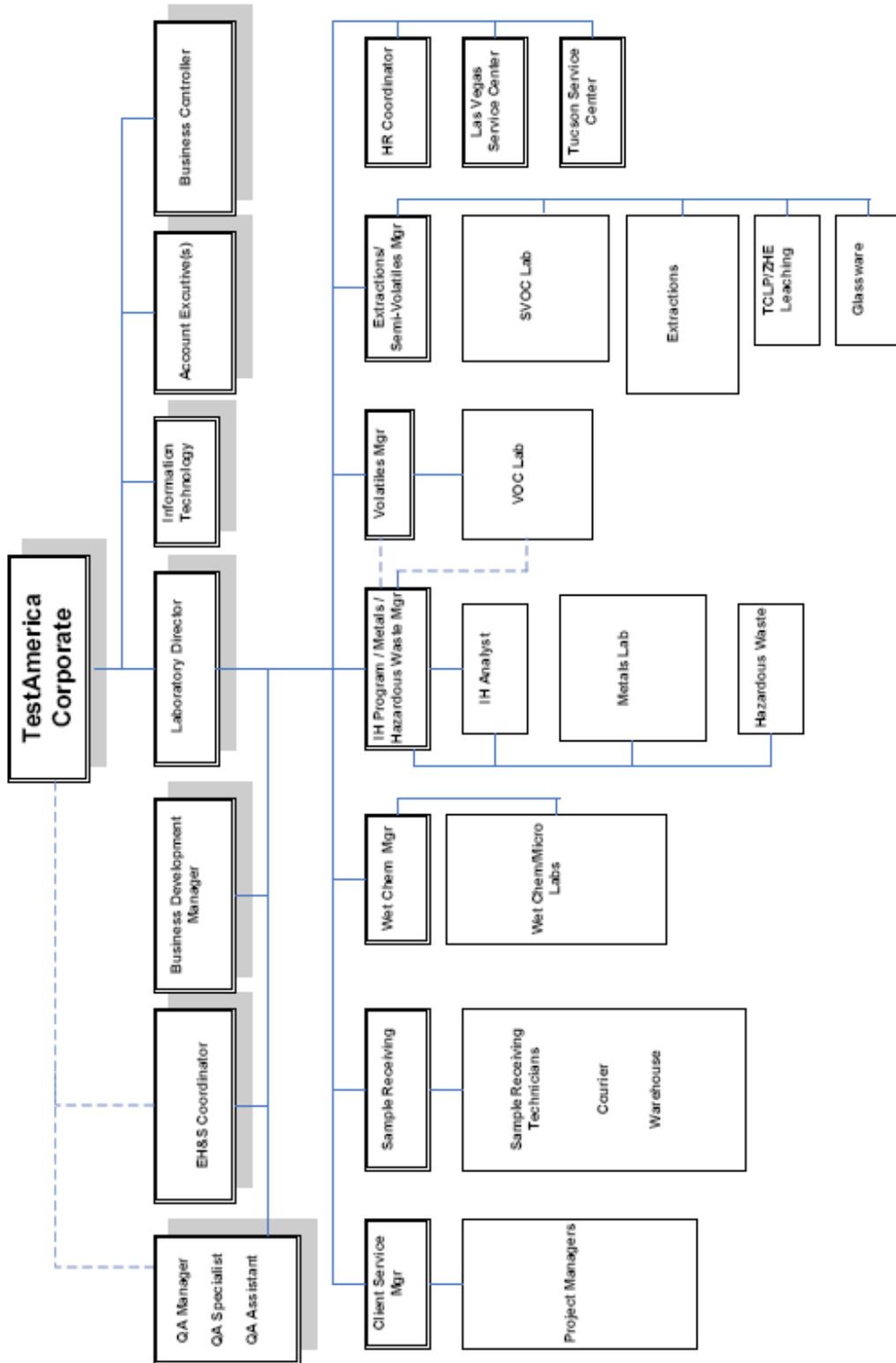
#### 4.3 Deputies

The following table defines who assumes the responsibilities of key personnel in their absence:

<b>Key Personnel</b>	<b>Deputy</b>
Laboratory Director	Client Services Manager
QA Manager	Laboratory Director and / or QA Specialist
Client Services Manager	Laboratory Director
Industrial Hygiene Program/Technical Manager	Client Services Manager
VOA Department Manager	VOAs Lead, SVOAs Manager and /or Laboratory Director
SVOA/Extractions Department Manager	SVOAs Lead, VOAs Manager and / or Laboratory Director
Wet Chemistry Department Manager	Wet Chem Lead and / or Laboratory Director
Metals Department Manager	SVOA Manger, Metals Lead, and / or Laboratory Director or CSM
Sample Receiving Department Manager	Sample Receiving Group Leader, Client Services Manager
Hazardous Waste Coordinator	Environmental Health & Safety Coordinator and / or Laboratory Director
Environmental Health & Safety Coordinator	Hazardous Waste Coordinator/ or Laboratory Director

Figure 4-1. Corporate and Laboratory Organization Charts





**Note:** An organizational chart with employee names is kept on file in the QA Department

## SECTION 5. QUALITY SYSTEM

### 5.1 Quality Policy Statement

It is TestAmerica's Policy to:

- ❖ Provide data of known quality to its clients by adhering to approved methodologies, regulatory requirements and the QA/QC protocols.
- ❖ Effectively manage all aspects of the laboratory and business operations by the highest ethical standards.
- ❖ Continually improve systems and provide support to quality improvement efforts in laboratory, administrative and managerial activities. TestAmerica recognizes that the implementation of a quality assurance program requires management's commitment and support as well as the involvement of the entire staff.
- ❖ Provide clients with the highest level of professionalism and the best service practices in the industry.
- ❖ Comply with the ISO/IEC 17025:2005(E) International Standard, the 2009 TNI Standard and to continually improve the effectiveness of the management system.

Every staff member at the laboratory plays an integral part in quality assurance and is held responsible and accountable for the quality of their work. It is, therefore, required that all laboratory personnel are trained and agree to comply with applicable procedures and requirements established by this document.

### 5.2 Ethics and Data Integrity

TestAmerica is committed to ensuring the integrity of its data and meeting the quality needs of its clients. The elements of TestAmerica's Ethics and Data Integrity Program include:

- An Ethics Policy (Corporate Policy No. CW-L-P-004 and Employee Ethics Statements).
- Ethics and Compliance Officers (ECOs).
- A Training Program.
- Self-governance through disciplinary action for violations.
- A Confidential mechanism for anonymously reporting alleged misconduct and a means for conducting internal investigations of all alleged misconduct. (Corporate SOP No. CW-L-S-002).
- Procedures and guidance for recalling data if necessary (Corporate SOP No. CW-L-S-002).
- Effective external and internal monitoring system that includes procedures for internal audits (Section 15).
- Produce results, which are accurate and include QA/QC information that meets client 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 to the extent and kinds of services available.
- Assert competency only for work for which adequate personnel and equipment are available and for which adequate preparation has been made.
- Promote the status of environmental laboratories, their employees, and the value of services rendered by them.

### **5.3 Quality System Documentation**

The laboratory's Quality System is communicated through a variety of documents.

- Quality Assurance Manual – Each laboratory has a lab-specific quality assurance manual.
- 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
- Laboratory QA/QC Policy Memorandums

#### **5.3.1 Order of Precedence**

In the event of a conflict or discrepancy between policies, the order of precedence is as follows:

- Corporate Quality Management Plan (CQMP)
- Corporate SOPs and Policies
- Laboratory QA/QC Policy Memorandum
- Laboratory Quality Assurance Manual (QAM)
- Laboratory SOPs and Policies
- Other (Work Instructions (WI), memos, flow charts, etc.)

Note: The laboratory has the responsibility and authority to operate in compliance with regulatory requirements of the jurisdiction in which the work is performed. Where the CQMP conflicts with those regulatory requirements, the regulatory requirements of the jurisdiction shall hold primacy. The laboratory's QAM shall take precedence over the CQMP in those cases.

## **5.4 QA/QC Objectives for the Measurement of Data**

Quality Assurance (QA) and Quality Control (QC) are activities undertaken to achieve the goal of producing data that accurately characterize the sites or materials that have been sampled. Quality Assurance is generally understood to be more comprehensive than Quality Control. Quality Assurance can be defined as the integrated system of activities that ensures that a product or service meets defined standards.

Quality Control is generally understood to be limited to the analyses of samples and to be synonymous with the term "*analytical quality control*". QC refers to the routine application of statistically based procedures to evaluate and control the accuracy of results from analytical measurements. The QC program includes procedures for estimating and controlling precision and bias and for determining reporting limits.

Request for Proposals (RFPs) and Quality Assurance Project Plans (QAPP) provide a mechanism for the client and the laboratory to discuss the data quality objectives in order to ensure that analytical services closely correspond to client needs. The client is responsible for developing the QAPP. In order to ensure the ability of the laboratory to meet the Data Quality Objectives (DQOs) specified in the QAPP, clients are advised to allow time for the laboratory to review the QAPP before being finalized. Additionally, the laboratory will provide support to the client for developing the sections of the QAPP that concern laboratory activities.

Historically, laboratories have described their QC objectives in terms of precision, accuracy, representativeness, comparability, completeness, selectivity and sensitivity (PARCCSS). For AIHA we add Bias and Measurement Uncertainty.

### **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.

### **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.

### **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.

### **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), interelement corrections (separation), use of matrix modifiers (separation), specific retention times (separation and identification), confirmations with different columns or detectors (separation and identification), specific wavelengths (identification), specific mass spectra (identification), specific electrodes (separation and identification), etc..

#### **5.4.7 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 precision and accuracy acceptability limits for performed analyses can be found in Element. This summary includes an effective date, is updated each time new limits are generated and are managed by the laboratory's QA department. Unless otherwise noted, limits within these tables are laboratory generated. Some acceptability limits are derived from published methods (US EPA methods and other regulatory methods) when they are required. Where method limits are not required, the laboratory has developed limits from evaluation of data from similar matrices. Criteria for development of control limits are contained in SOP PE-QAD-001 Control Charts and Statistical Process Control and/or Section 24

#### **5.6 Statistical Quality Control**

Statistically-derived precision and accuracy limits are required by selected methods (such as SW-846) and programs [such as Arizona Department of Health Services (ADHS)]. The laboratory routinely utilizes statistically-derived limits to evaluate method performance and determine when corrective action is appropriate. The analysts are instructed to use the current limits, dated and approved by the Department Manager and QA Manager, The limits are entered into the Laboratory Information Management System (LIMS). The test's limits associated with data are archived in LIMS.

If a method defines the QC limits, the method limits are used. On occasion, a client may request contract-specified limits for a specific project. These limits may be used if they are equal to or more restrictive than those specified by the method.

If a method requires the generation of historical limits, the lab develops such limits from recent data in the QC database of the LIMS following the guidelines described in Section 24. All calculations and limits are documented and dated when approved and effective. On occasion, a client requests contract-specified limits for a specific project.

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**

QC charts are generated as part of statistical control (see SOP PE-QAD-001). The QA Manager and Department Manager evaluate these to determine if adjustments need to be made or for corrective actions to methods. All findings are documented and kept on file. The charts are available for analyst review on the shared laboratory directory.

## **5.7 Quality System Metrics**

In addition to the QC parameters discussed above, the entire Quality System is evaluated on a monthly basis through the use of specific metrics (refer to Section 16). These metrics are used to drive continuous improvement in the laboratory's Quality System.

## **SECTION 6. DOCUMENT CONTROL**

### **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:

- Laboratory Quality Assurance Manual
- Laboratory Standard Operating Procedures (SOP)
- Laboratory Policies
- Work Instructions and Forms
- Corporate Policies and Procedures distributed outside the intranet

Corporate Quality posts Corporate Manuals, SOPs, Policies, Work Instructions, White Papers and Training Materials on the company intranet site. These Corporate documents are only considered controlled when they are read on the intranet site. Printed copies are considered uncontrolled unless the laboratory physically distributes them as controlled documents. A detailed description of the procedure for issuing, authorizing, controlling, distributing, and archiving Corporate documents is found in Corporate SOP No. CW-Q-S-001, Corporate Document Control and Archiving. The laboratory's internal document control procedure is defined in SOP PE-QAD-010 Document Control.

The laboratory QA Department also maintains access to various references and document sources integral to the operation of the laboratory. This includes reference methods and regulations. Instrument manuals (hard or electronic copies) are also maintained by the laboratory.

The laboratory maintains control of records for raw analytical data and supporting records such as internal audit reports and responses, logbooks, standard logs, training files, MDL studies, Proficiency Testing (PT) studies, certifications and related correspondence, and corrective action reports. Raw analytical data consists of bound logbooks, instrument printouts, any other notes, magnetic media, electronic data and final reports.

### **6.2 Document Approval and Issue**

The pertinent elements of a document control system for each document include a unique document title and number, pagination, the total number of pages of the item or an 'end of document' page, the effective date, revision number and the laboratory's name. The QA personnel are responsible for the maintenance of this system.

Controlled documents are authorized by the QA Department and other management personnel. In order to develop a new document, a Technical/Department Manager submits an electronic draft to the QA Department for suggestions and approval before use. Spreadsheets used for calculations and data evaluation must be verified to be accurate and locked down prior to approval. Upon approval, QA personnel add the identifying version information to the document and retains that document as the official document on file. That document is then provided to all applicable operational units (may include electronic access). Controlled documents are identified as such and records of their distribution are kept by the QA Department. Document control may be achieved by either electronic or hardcopy distribution.

The QA Department maintains a list of the official versions of controlled documents.

Quality System Policies and Procedures will be reviewed at a minimum of every year for drinking water and AIHA methods, and every two years for all other methods and are revised as appropriate. Changes to documents occur when a procedural change warrants.

### **6.3 Procedures for Document Control Policy**

For changes to the QA Manual, refer to SOP PE-QAD-010 Document Control. Previous revisions and back-up data are stored by the QA Department. Electronic copies are stored on the Public server in the QA folder for the applicable revision, accessible to all Phoenix employees.

For changes to SOPs, refer to SOP PE-QAD-014 Creation and Maintenance of SOPs. The SOP identified above also defines the process of changes to SOPs.

Forms, worksheets, work instructions and information are organized and maintained by the QA Department. A table of contents and electronic versions are kept on the QA department server. The procedure for the care of these documents is in SOP PE-QAD-010 Document Control.

### **6.4 Obsolete Documents**

All invalid or obsolete documents are removed, or otherwise prevented from unintended use. The laboratory has specific procedures as described above to accomplish this. At least one copy of the obsolete document is archived according to SOP PE-QAD-014 Creation and Maintenance of SOPs and SOP PE-QAD-010 Document Control.

## **SECTION 7. SERVICE TO THE CLIENT**

### **7.1 Overview**

The laboratory has established procedures for the review of work requests and contracts, oral or written. The procedures include evaluation of the laboratory's capability and resources to meet the contract's requirements within the requested time period. All requirements, including the methods to be used, must be adequately defined, documented and understood. For many environmental sampling and analysis programs, testing design is site or program specific and does not necessarily "fit" into a standard laboratory service or product. It is the laboratory's intent to provide both standard and customized environmental laboratory services to our clients.

A thorough review of technical and QC requirements contained in contracts is performed to ensure project success. The appropriateness of requested methods, and the lab's capability to perform them must be established. Projects, proposals and contracts are reviewed for adequately defined requirements and the laboratory's capability to meet those requirements. Alternate test methods that are capable of meeting the clients' requirements may be proposed by the lab. A review of the lab's capability to analyze non-routine analytes is also part of this review process.

All projects, proposals and contracts are reviewed for the client's requirements in terms of compound lists, test methodology requested, sensitivity (detection and reporting levels), accuracy, and precision requirements (% Recovery and RPD). The reviewer ensures that the laboratory's test methods are suitable to achieve these requirements and that the laboratory holds the appropriate certifications and approvals to perform the work. The laboratory and any potential subcontract laboratories must be certified, as required, for all proposed tests.

The laboratory must determine if it has the necessary physical, personnel and information resources to meet the contract, and if the personnel have the expertise needed to perform the testing requested. Each proposal is checked for its impact on the capacity of the laboratory's equipment and personnel. As part of the review, the proposed turnaround time will be checked for feasibility.

Electronic or hard copy deliverable requirements are evaluated against the laboratory's capacity for production of the documentation.

If the laboratory cannot provide all services but intends to subcontract such services, whether to another TestAmerica facility or to an outside firm, this will be documented and discussed with the client prior to contract approval. (Refer to Section 8 for Subcontracting Procedures.)

The laboratory informs the client of the results of the review if it indicates any potential conflict, deficiency, lack of accreditation, or inability of the lab to complete the work satisfactorily. Any discrepancy between the client's requirements and the laboratory's capability to meet those requirements is resolved in writing before acceptance of the contract. It is necessary that the contract be acceptable to both the laboratory and the client. Amendments initiated by the client and/or TestAmerica, are documented in writing.

All contracts, QAPPs, Sampling and Analysis Plans (SAPs), contract amendments, and documented communications become part of the project record. The same contract review process used for the initial review is repeated when there are amendments to the original contract by the client, and the participating personnel are informed of the changes.

## **7.2 Review Sequence and Key Personnel**

Appropriate personnel will review the work request at each stage of evaluation.

For routine projects and other simple tasks, a review by the Project Manager (PM) is considered adequate. The PM confirms that the laboratory has any required certifications, that it can meet the clients' data quality and reporting requirements and that the lab has the capacity to meet the clients turn around needs. It is recommended that, where there is a sales person assigned to

the account, an attempt should be made to contact that sales person to inform them of the incoming samples.

For new, complex or large projects, the proposed contract is given to the National Account Director or other appropriate personnel, who will decide which lab will receive the work based on the scope of work and other requirements, including certification, testing methodology, and available capacity to perform the work. The contract review process is outlined in TestAmerica's Corporate SOP CA-L-P-002, Contract Compliance Policy.

This review encompasses all facets of the operation. The scope of work is distributed to the appropriate personnel, as needed based on scope of contract, to evaluate all of the requirements shown above (not necessarily in the order below):

- Legal & Contracts Director
- General Manager
- The Laboratory Client Services Manager/Business Development Manager
- Laboratory and/or Corporate Technical/Department Managers
- Laboratory and/or Corporate Information Technology Managers/Directors
- Account Executives
- Laboratory and/or Corporate Quality Assurance Personnel
- Laboratory and/or Corporate Environmental Health and Safety Managers/Directors
- The Laboratory Director reviews the formal laboratory quote and makes final acceptance for their facility.

The National Account Director, Legal Contracts Director, or local account representative then submits the final proposal to the client.

In the event that one of the above personnel is not available to review the contract, his or her back-up will fulfill the review requirements.

The Legal & Contracts Director maintains copies of all signed contracts. The Business Development Manager or the Project Manager maintains the local copies of the contracts.

### **7.3 Documentation**

Appropriate records are maintained for every contract or work request. All stages of the contract review process are documented and include records of any significant changes. Those records are maintained locally as needed.

The contract will be distributed to and maintained by the appropriate sales/marketing personnel. A copy of the contract and formal quote will be filed with the laboratory Project Manager (PM).

Records are maintained of pertinent discussions with a client relating to the client's requirements or the results of the work during the period of execution of the contract. The PM keeps a phone log of conversations with the client. Client correspondence and internal communications regarding projects are kept in the project file or stored electronically.

### **7.3.1 Project-Specific Quality Planning**

Communication of contract specific technical and QC criteria is an essential activity in ensuring the success of site specific testing programs. To achieve this goal, the laboratory assigns a PM to each client. It is the PM's responsibility to ensure that project specific technical and QC requirements are effectively evaluated and communicated to the laboratory personnel before and during the project. QA Department involvement may be needed to assist in the evaluation of custom QC requirements.

PMs are the primary client contact and they ensure resources are available to meet project requirements. Although PM's do not have direct reports or staff in production, they coordinate opportunities and work with laboratory management and supervisory staff to ensure available resources are sufficient to perform work for the client's project. Project management is positioned between the client and laboratory resources.

Prior to working 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 status 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 or contract addendum, which has been signed by both parties.

Such changes are also communicated verbally to the laboratory during status meetings. Such changes are updated to the project notes and are introduced to the managers at these meetings. The laboratory staff is then introduced to the modified requirements via the PM or the individual laboratory Department Manager. After the modification is implemented into the laboratory process, documentation of the modification is made in the case narrative of the data report(s).

The laboratory strongly encourages client visits to the laboratory and for formal/informal information sharing session with employees in order to effectively communicate ongoing client needs as well as project specific details for customized testing programs.

### **7.4 Special Services**

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. It is the laboratory's goal to meet all client requirements in addition to statutory and regulatory requirements. The laboratory has procedures to ensure confidentiality to clients (Section 15 and 25).

Note: ISO/IEC 17025 states that a laboratory “shall afford clients or their representatives’ cooperation to clarify the client’s request”.

The laboratory’s standard procedures for reporting data are described in Section 25. Special services are also available and provided upon request. These services include:

- Reasonable access for our clients or their representatives to the relevant areas of the laboratory for the witnessing of tests performed for the client.
- Assist client-specified third party data validators as specified in the client’s contract.
- Supplemental information pertaining to the analysis of their samples. Note: An additional charge may apply for additional data/information that was not requested prior to the time of sample analysis or previously agreed upon.

### **7.5 Client Communication**

Project Managers are the primary communication link to the clients. They shall inform their clients of any delays in project completion as well as any non-conformances in either sample receipt or sample analysis. Project Management will maintain ongoing client communication throughout the entire client project.

Technical Manager/Department Managers are available to discuss any technical questions or concerns that the client may have.

### **7.6 Reporting**

The laboratory works with our clients to produce any special communication reports required by the contract.

### **7.7 Client Surveys**

The laboratory assesses both positive and negative client feedback. The results are used to improve overall laboratory quality and client service. TestAmerica’s Sales and Marketing teams periodically develops lab and client specific surveys to assess client satisfaction.

## **SECTION 8. SUBCONTRACTING OF TESTS**

### **8.1 Overview**

For the purpose of this quality manual, the phrase subcontract laboratory refers to a laboratory external to the TestAmerica laboratories. The phrase “work sharing” refers to internal transfers of samples between the TestAmerica laboratories. The term outsourcing refers to the act of subcontracting tests.

When contracting with our clients, the laboratory makes commitments regarding the services to be performed and the data quality for the results to be generated. When the need arises to outsource testing for our clients because project scope, changes in laboratory capabilities, capacity or unforeseen circumstances, we must be assured that the subcontractors or work sharing laboratories understand the requirements and will meet the same commitments we

have made to the client. Refer to TestAmerica's Corporate SOP's on Subcontracting Procedures (CA-L-S-002) and the Work Sharing Process (CA-C-S-001).

When outsourcing analytical services, the laboratory will assure, to the extent necessary, that the subcontract or work sharing laboratory maintains a program consistent with the requirements of this document, the requirements specified in State/TNI/AIHA/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-TNI/AIHA or regulatory accredited work where required.

Project Managers (PMs), Customer Service Manager (CSM), or Account Executives 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 (e.g, USDA) or contracts -may require notification prior to placing such work.

## **8.2 Qualifying and Monitoring Subcontractors**

Whenever a PM, Account Executive or CSM becomes aware of a client requirement or laboratory need where samples must be outsourced to another laboratory, the other laboratory(s) shall be selected based on the following:

- The first priority is to attempt to place the work in a qualified TestAmerica laboratory;
- Firms specified by the client for the task (Documentation that a subcontractor was designated by the client must be maintained with the project file. This documentation can be as simple as placing a copy of an e-mail from the client in the project folder);
- Firms listed as pre-qualified and currently under a subcontract with TestAmerica: A listing of all approved subcontracting laboratories is available on the TestAmerica intranet site. Supporting documentation is maintained by corporate offices and by the TestAmerica laboratory originally requesting approval of the subcontract lab. Verify necessary accreditation, where applicable, (e.g., on the subcontractors TNI, A2LA accreditation or State Certification).
- Firms identified in accordance with the company's Small Business Subcontracting program as small, women-owned, veteran-owned and/or minority-owned businesses;
- TNI or AIHA accredited laboratories.
- In addition, the firm must hold the appropriate certification/accreditation to perform the work required. For TNI and AIHA accreditation, this would include accreditation to the same Field of Testing.

All TestAmerica laboratories are pre-qualified for work sharing provided they hold the appropriate accreditations, can adhere to the project/program requirements, and the client approved sending samples to that laboratory. The client must provide acknowledgement that

the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented). The originating laboratory is responsible for communicating all technical, quality, and deliverable requirements as well as other contract needs. (Corporate SOP No. CA-C-S-001, Work Sharing Process).

When the potential sub-contract laboratory has not been previously approved, Account Executives or PMs may nominate a laboratory as a subcontractor based on need. The decision to nominate a laboratory must be approved by the Laboratory Director. The Laboratory Director requests that the QA Manager begin the process of approving the subcontract laboratory as outlined in Corporate SOP CA-L-S-002, Subcontracting Procedures. The client must provide acknowledgement that the samples can be sent to that facility (an e-mail is sufficient documentation or if acknowledgement is verbal, the date, time, and name of person providing acknowledgement must be documented).

**8.2.1** Once the appropriate accreditation and legal information is received by the laboratory, it is evaluated for acceptability (where applicable) and forwarded to Corporate Contracts for formal contracting with the laboratory. They will add the lab to the approved list on the intranet site and notify the finance group for JD Edwards.

**8.2.2** The client will assume responsibility for the quality of the data generated from the use of a subcontractor they have requested the lab to use. The qualified subcontractors on the intranet site are known to meet minimal standards. TestAmerica does not certify laboratories. The subcontractor is on our approved list and can only be recommended to the extent that we would use them.

**8.2.3** The status and performance of qualified subcontractors will be monitored periodically by the Corporate Contracts and/or Quality Departments. Any problems identified will be brought to the attention of TestAmerica's Corporate Finance or Corporate Quality personnel.

- Complaints shall be investigated. Documentation of the complaint, investigation and corrective action will be maintained in the subcontractor's file on the intranet site. Complaints are posted using the Vendor Performance Report.
- Information shall be updated on the intranet when new information is received from the subcontracted laboratories.
- Subcontractors in good standing will be retained on the intranet listing. The QA Manager will notify all TestAmerica laboratories, Corporate Quality and Corporate Contracts if any laboratory requires removal from the intranet site. This notification will be posted on the intranet site and e-mailed to all Laboratory Directors, QA Managers and Sales Personnel.

### **8.3 Oversight and Reporting**

The PM, CSM or Account Executive 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, CSM or Account Executive 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 the Client Approved Subcontracted Sample Form, PX-PDM-012 and the form is retained in the project folder. The form is not required if the need to subcontract the analysis has been identified in the project quote. For TestAmerica laboratories, certifications can be viewed on the company's TotalAccess Database.

The Sample Control department is responsible for ensuring compliance with QA requirements and applicable shipping regulations when shipping samples to a subcontracted laboratory.

All subcontracted samples must be accompanied by a TestAmerica Chain of Custody (COC). A copy of the original COC sent by the client must also be included with all samples workshared within TestAmerica. Client CoCs are only forwarded to external subcontractors when samples are shipped directly from the project site to the subcontractor lab. Under routine circumstances, client CoCs are not provided to external subcontractors.

Through communication with the subcontracted laboratory, the PM monitors the status of the subcontracted analyses, facilitates successful execution of the work, and ensures the timeliness and completeness of the analytical report.

Non-TNI, non-ADHS or non-AIHA accredited work must be identified in the subcontractor's report as appropriate. If accreditations are not required, the report does not need to include this information.

Reports submitted from subcontractor laboratories are not altered and are included in their original form in the final project report. This clearly identifies the data as being produced by a subcontractor facility. If subcontract laboratory data is incorporated into the laboratories EDD (i.e., imported), the report must explicitly indicate which lab produced the data for which methods and samples.

**Note:** The results submitted by a TestAmerica work sharing laboratory may be transferred electronically and the results reported by the TestAmerica work sharing lab are identified on the final report. The report must explicitly indicate which lab produced the data for which methods and samples. The final report must include a copy of the completed COC for all work sharing reports.

#### **8.4 Contingency Planning**

The Laboratory Director may waive the full qualification of a subcontractor process temporarily to meet emergency needs; however, this decision & justification must be documented in the project files, and the 'Purchase Order Terms And Conditions For Subcontracted Laboratory Services' must be sent with the samples and Chain-of-Custody. In the event this provision is utilized, the laboratory (e.g., PM) will be required to verify and document the applicable accreditations of the subcontractor. All other quality and accreditation requirements will still be applicable, but the subcontractor need not have signed a subcontract with TestAmerica at this



## **SECTION 9. PURCHASING SERVICES AND SUPPLIES**

### **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, which may affect quality, all purchases from specific vendors are approved by a member of the supervisory or management staff. Capital expenditures are made in accordance with TestAmerica's Corporate Controlled Purchases Procedure, SOP No. CW-F-S-007.

Contracts will be signed in accordance with TestAmerica's Corporate Authorization Matrix Policy, Policy No. CW-F-P-002. Request for Proposals (RFP's) will be issued where more information is required from the potential vendors than just price. Process details are available in TestAmerica's Corporate Procurement and Contracts Policy (Policy No. CW-F-P-004). RFP's allow TestAmerica to determine if a vendor is capable of meeting requirements such as supplying all of the TestAmerica facilities, meeting required quality standards and adhering to necessary ethical and environmental standards. The RFP process also allows potential vendors to outline any additional capabilities they may offer.

### **9.2 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**

Purchasing guidelines for equipment and reagents must meet the requirements of the specific method and testing procedures for which they are being purchased. Solvents and acids are pre-tested in accordance with TestAmerica's Corporate SOP on Solvent & Acid Lot Testing & Approval, SOP No. CA-Q-S-001.

#### **9.3.1 Purchasing**

Chemical reagents, solvents, glassware, and general supplies are ordered as needed to maintain sufficient quantities on hand. Materials used in the analytical process must be of a known quality. The wide variety of materials and reagents available makes it advisable to specify recommendations for the name, brand, and grade of materials to be used in any determination. This information is contained in the method SOP.

The requisitioning department employee will complete an ordering template found on the shared server. Once completed the template should be saved in the proper folder with the employee's initials and date. This will be forwarded to the Administrative personnel in charge of ordering who will complete a General Requisition in JD Edwards. Alternatively, some departments complete the General Requisition in JD Edwards directly. The Lab Director will approve the

requisition via the orders awaiting approval application in JDE. Properly approved requisitions are generated into purchase orders and are procured by the Corporate Purchasing Coordinator.

### 9.3.2 Receiving

It is the responsibility of warehouse personnel to receive the shipment. They must also document date the material when received and compare the information on the label or packaging to the original order to ensure that the purchase meets the quality level specified. A unique tracking identifier is assigned at this time. The laboratory department receiving the item confirms that the quality of the item received meets the level specified. Material Safety Data Sheets (MSDSs) are available online through the Company's intranet website. Anyone may review these for relevant information on the safe handling and emergency precautions of on-site chemicals. Any MSDS should be given to EHS for review. The intranet is checked to determine if the MSDS is already available. If it is not an electronic copy of the MSDS is sent to corporate EHS where it is added to the Company's intranet.

### 9.3.3 Specifications

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, analytical reagent grade will 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 unless the conditions outlined below are followed. If expiration dates are not provided, the laboratory may contact the manufacturer to determine an expiration date.

The laboratory assumes a five year expiration date on inorganic dry chemicals and solvents unless noted otherwise by the manufacturer or by the reference source method. Chemicals/solvents should not be used past the manufacturers' or SOPs expiration date unless 'verified' (refer to item 3 listed below).

- An expiration date **cannot** be extended if the dry chemical/solvent is discolored or appears otherwise physically degraded, the dry chemical/solvent must be discarded.
- Expiration dates can be extended if the dry chemical/solvent 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/solvent is used for the preparation of standards, the expiration dates can be extended 6 months if the dry chemical/solvent is compared to an unexpired independent source in performing the method and the performance of the dry chemical/solvent is found to be satisfactory. The comparison must show that the dry chemical/solvent meets CCV limits. The comparison studies are maintained in the QA office.

Note: The five year expiration date applies to all Industrial Hygiene standards that are considered 'neat'.

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. To prevent a tank from going to dryness or introducing potential impurities, the pressure should be closely watched as it decreases to approximately 15% of the original reading, at which point it should be replaced. For example, a standard sized laboratory gas cylinder containing 3,000 psig of gas should be replaced when it drops to approximately 500 psig. The quality of the gases must meet method or manufacturer specification or be of a grade that does not cause any analytical interference. Gas cylinders are tracked by manufacturer, pressure, and lot number.

Water used in the preparation of standards or reagents must have a specific conductivity of less than 1-  $\mu\text{mho/cm}$  (or specific resistivity of greater than 1.0 megohm-cm) at 25°C. The specific conductivity is checked and recorded daily. If the water's specific conductivity is greater than the specified limit, the Facility Manager and appropriate Technical/Department Managers must be notified immediately in order to notify all departments, decide on cessation (based on intended use) of activities, and make arrangements for correction.

The laboratory may purchase reagent grade (or other similar quality) water for use in the laboratory. This water must be certified "clean" by the supplier for all target analytes or otherwise verified by the laboratory prior to use. This verification is documented.

Standard lots are verified before first time use if the laboratory switches manufacturers or has historically had a problem with the type of standard.

Purchased bottleware used for sampling must be certified clean and the certificates must be maintained. If uncertified sampling bottleware is purchased, all lots must be verified clean prior to use. This verification must be maintained.

Records of manufacturer's certification and traceability statements are entered into LIMS. They may also be maintained in files or binders in each laboratory section. These records include date of receipt, lot number (when applicable), and expiration date (when applicable).

#### **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. Storage conditions are per the Corporate Environmental Health & Safety Manual (Corp. Doc. No. CW-E-M-001) and method SOPs or manufacturer instructions.

#### **9.4 Purchase of Equipment / Instruments / Software**

When a new piece of equipment is needed, either for additional capacity or for replacing inoperable equipment, the analyst or supervisor makes a supply request to the Technical or Department Manager or and/or the Laboratory Director. If they agree with the request, the procedures outlined in TestAmerica's Corporate Policy No. CA-T-P-001, Qualified Products List, are followed. A decision is made as to which piece of equipment can best satisfy the requirements. The appropriate written requests are completed and purchasing places the order.

Upon receipt of a new or used piece of equipment, an identification name is assigned and added to the equipment list. IT must also be notified so that they can synchronize the instrument for back-ups. Its capability is assessed to determine if it is adequate or not for the specific application. For instruments, a calibration curve is generated, followed by MDLs, Demonstration of Capabilities (DOCs), and other relevant criteria (refer to Section 19). For software, its operation must be deemed reliable and evidence of instrument verification must be retained by the IT Department. Software certificates supplied by the vendors are filed with the local IT Department. The manufacturer's operation manual is retained at the bench.

## **9.5 Services**

Service to analytical instruments (except analytical balances) is performed on an as needed basis. Routine preventative maintenance is discussed in Section 20. The need for service is determined by analysts and/or Technical/Department Managers. The service providers that perform the services are approved by the Technical Manager/Department Manager/Laboratory Director.

## **9.6 Suppliers**

TestAmerica selects vendors through a competitive proposal / bid process, strategic business alliances or negotiated vendor partnerships (contracts). This process is defined in the Corporate Finance documents on Vendor Selection (SOP No. CW-F-S-018) and Procurement & Contracts Policy (Policy No. CW-F-P-004). The level of control used in the selection process is dependent on the anticipated spending amount and the potential impact on TestAmerica business. Vendors that provide test and measuring equipment, solvents, standards, certified containers, instrument related service contracts or subcontract laboratory services shall be subject to more rigorous controls than vendors that provide off-the-shelf items of defined quality that meet the end use requirements. The JD Edwards purchasing system includes all suppliers/vendors that have been approved for use.

Evaluation of suppliers is accomplished by ensuring the supplier ships the product or material ordered and that the material is of the appropriate quality. This is documented by signing off on packing slips or other supply receipt documents. The purchasing documents contain the data that adequately describe the services and supplies ordered.

Any issues of vendor performance are to be reported immediately by the laboratory staff to the Corporate Purchasing Group by completing a Vendor Performance Report.

The Corporate Purchasing Group will work through the appropriate channels to gather the information required to clearly identify the problem and will contact the vendor to report the problem and to make any necessary arrangements for exchange, return authorization, credit, etc.

As deemed appropriate, the Vendor Performance Reports will be summarized and reviewed to determine corrective action necessary, or service improvements required by vendors

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.

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.

## **SECTION 10. COMPLAINTS**

### **10.1 Overview**

The laboratory considers an effective client complaint handling processes to be of significant business and strategic value. Listening to and documenting client concerns captures 'client knowledge' that enables our operations to continually improve processes and client satisfaction. An effective client complaint handling process also provides assurance to the data user that the laboratory will stand behind its data, service obligations and products.

A client complaint is any expression of dissatisfaction with any aspect of our business services (e.g., communications, responsiveness, data, reports, invoicing and other functions) expressed by any party, whether received verbally or in written form. Client inquiries, complaints or noted discrepancies are documented, communicated to management, and addressed promptly and thoroughly.

The laboratory has procedures for addressing both external and internal complaints with the goal of providing satisfactory resolution to complaints in a timely and professional manner.

The nature of the complaint is identified, documented and investigated, and an appropriate action is determined and taken. In cases where a client complaint indicates that an established policy or procedure was not followed, the QA Department must evaluate whether a special audit must be conducted to assist in resolving the issue. A written confirmation or letter to the client, outlining the issue and response taken is recommended as part of the overall action taken.

The process of complaint resolution and documentation utilizes the procedures outlined in Section 12 (Corrective Actions) and SOP PE-QAD-027 Procedures to Address Customer Complaints. It is documented following laboratory SOP PE-PMD-002 Project Management Communication and Documentation, including entry into LIMS.

## **10.2 External Complaints**

An employee that receives a complaint initiates the complaint resolution process by first documenting the complaint according to SOPs PE-QAD-027, Procedures to Address Customer Complaints, and PE-PMD-002 Project Management Communication and Documentation.

Complaints fall into two categories: correctable and non-correctable. An example of a correctable complaint would be one where a report re-issue would resolve the complaint. An example of a non-correctable complaint would be one where a client complains that their data was repeatedly late. Non-correctable complaints should be reviewed for preventive action measures to reduce the likelihood of future occurrence and mitigation of client impact.

The general steps in the complaint handling process are:

- Receiving and Documenting Complaints
- Complaint Investigation and Service Recovery
- Process Improvement

The laboratory shall inform the initiator of the complaint of the results of the investigation and the corrective action taken, if any.

## **10.3 Internal Complaints**

Internal complaints include, but are not limited to: errors and non-conformances, training issues, internal audit findings, and deviations from methods. Corrective actions may be initiated by any staff member who observes a nonconformance and shall follow the procedures outlined in Section 12. In addition, Corporate Management, Sales and Marketing and IT may initiate a complaint by contacting the laboratory or through the corrective action system described in Section 12.

## **10.4 Management Review**

The number and nature of client complaints is reported by the QA Manager to the laboratory and QA Director in the QA Monthly report. Monitoring and addressing the overall level and nature of client complaints and the effectiveness of the solutions is part of the Annual Management Review (Section 16).

# **SECTION 11. CONTROL OF NON-CONFORMING WORK**

## **11.1 Overview**

When data discrepancies are discovered or deviations and departures from laboratory SOPs, policies and/or client requests have occurred, corrective action is taken immediately. First, the laboratory evaluates the significance of the nonconforming work. Then, a corrective action plan is initiated based on the outcome of the evaluation. If it is determined that the nonconforming work is an isolated incident, the plan could be as simple as adding a qualifier to the final results and/or making a notation in the case narrative. If it is determined that the nonconforming work is a systematic or improper practices issue, the corrective action plan could include a more in depth

investigation and a possible suspension of an analytical method. In all cases, the actions taken are documented using the laboratory's corrective action system (refer to Section 12).

Due to the frequently unique nature of environmental samples, sometimes departures from documented policies and procedures are needed. When an analyst encounters such a situation, the problem is presented to the Technical/Department Manager for resolution. The Manager may elect to discuss it with the Laboratory Director and/or the QA Manager or have a representative contact the client to decide on a logical course of action. Once an approach is agreed upon, the analyst documents it in the analytical data. This information can then be supplied to the client in the form of a footnote or a case narrative with the report.

Project Management may encounter situations where a client may request that a special procedure be applied to a sample that is not standard lab practice. Based on a technical evaluation, the lab may accept or opt to reject the request based on technical or ethical merit. An example might be the need to report a compound that the lab does not normally report. The lab would not have validated the method for this compound following the procedures in Section 19. The client may request that the compound be reported based only on the calibration. Such a request would need to be approved by the Technical/Department Director and QA Manager, documented and included in the project folder. Deviations must also be noted on the final report with a statement that the compound is not reported in compliance with TNI (or the analytical method) requirements and the reason. Data being reported to a non- TNI state would need to note the change made to how the method is normally run.

## **11.2 Responsibilities and Authorities**

TestAmerica's Corporate SOP entitled Internal Investigation of Potential Data Discrepancies and Determination for Data Recall (SOP No. CW-L-S-002), outlines the general procedures for the reporting and investigation of data discrepancies and alleged incidents of misconduct or violations of TestAmerica's data integrity policies as well as the policies and procedures related to the determination of the potential need to recall data.

Under certain circumstances, the Laboratory Director, a Technical/Department Manager or a member of the QA team may authorize departures from documented procedures or policies. The departures may be a result of procedural changes due to the nature of the sample; a one-time procedure for a client; QC failures with insufficient sample to reanalyze, etc.. In most cases, the client will be informed of the departure prior to the reporting of the data. Any departures must be well documented using the laboratory's corrective action procedures. This information may also be documented in logbooks and/or data review checklists as appropriate. Any impacted data must be referenced in a case narrative and/or flagged with an appropriate data qualifier.

Any misrepresentation or possible misrepresentation of analytical data discovered by any laboratory staff member must be reported to facility Senior Management within 24-hours. The Senior Management staff is comprised of the Laboratory Director, the QA Manager, the Client Services Manager and the Department Managers. 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), the Director of Quality & Client Advocacy and the laboratory's Quality Director within 24 hours of discovery.

Whether an inaccurate result was reported due to calculation or quantitation errors, data entry errors, improper practices, or failure to follow SOPs, the data must be evaluated to determine the possible effect.

The Laboratory Director, QA Manager, ECOs, Corporate Quality, General Managers and the Quality Directors have the authority and responsibility to halt work, withhold final reports, or suspend an analysis for due cause as well as authorize the resumption of work. Any employee has the right to stop their work if they feel the quality may be compromised or cannot be completed as required.

### **11.3 Evaluation of Significance and Actions Taken**

For each nonconforming issue reported, an evaluation of its significance and the level of management involvement needed is made. This includes reviewing its impact on the final data, whether or not it is an isolated or systematic issue, and how it relates to any special client requirements.

TestAmerica's Corporate Data Investigation & Recall Procedure (SOP No. CW-L-S-002) distinguishes between situations when it would be appropriate for laboratory management to make the decision on the need for client notification (written or verbal) and data recall (report revision) and when the decision must be made with the assistance of the ECO's and Corporate Management. Laboratory level decisions are documented and approved using the laboratory's standard nonconformance/corrective action reporting in lieu of the data recall determination form contained in TestAmerica's Corporate SOP No. CW-L-S-002.

### **11.4 Prevention of Nonconforming Work**

If it is determined that the nonconforming work could recur, further corrective actions must be made following the laboratory's corrective action system. Periodically as defined by the laboratory's preventive action schedule, the QA Department evaluates non-conformances to determine if any nonconforming work has been repeated multiple times. If so, the laboratory's corrective action process may be followed.

### **11.5 Method Suspension / Restriction (Stop Work Procedures)**

In some cases, it may be necessary to suspend/restrict the use of a method or target compound which constitutes significant risk and/or liability to the laboratory. Suspension/restriction procedures can be initiated by any of the persons noted in Section 11.2, Paragraph 5.

Prior to method suspension/restriction, confidentiality will be respected, and the problem with the required corrective and preventive action will be stated in writing and presented to the Laboratory Director.

The Laboratory Director shall arrange for the appropriate personnel to meet with the QA Manager as needed. This meeting shall be held to confirm that there is a problem, that suspension/restriction of the method is required and will be concluded with a discussion of the steps necessary to bring the method/target or test fully back on line. In some cases, that may not be necessary if all appropriate personnel have already agreed there is a problem and there is agreement on the steps needed to bring the method, target or test fully back on line.

The QA Manager will also initiate a corrective action report as described in Section 12 if one has not already been started. A copy of any meeting notes and agreed upon steps should be faxed or e-mailed by the laboratory to the appropriate General Manager and member of Corporate QA. This fax/e-mail acts as notification of the incident.

After suspension/restriction, the lab will hold all reports to clients pending review. No faxing, mailing or distributing through electronic means may occur. The report must not be posted for viewing on the internet. It is the responsibility of the Laboratory Director to hold all reporting and to notify all relevant laboratory personnel regarding the suspension/restriction (e.g., Project Management, Log-in, etc...). Clients will NOT generally be notified at this time. Analysis may proceed in some instances depending on the non-conformance issue.

Within 72 hours, the QA Manager will determine if compliance is now met and reports can be released, OR determine the plan of action to bring work into compliance, and release work. A team, with all principals involved (Laboratory Director, Technical/ Department Manager, QA Manager, Client Services Manager) can devise a start-up plan to cover all steps from client notification through compliance and release of reports. Project Management, and the Directors of Client Services and Sales and Marketing must be notified if clients must be notified or if the suspension/restriction affects the laboratory's ability to accept work. The QA Manager must approve start-up or elimination of any restrictions after all corrective action is complete. This approval is given by final signature on the completed corrective action report.

## **SECTION 12. CORRECTIVE ACTION**

### **12.1 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. Non-conformance events and corrective actions are documented using Corrective Action Reports (CAR) (refer to Figure 12-1).

### **12.2 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(s) for investigating.
- Resolve non-conformance events and assign responsibility for any required corrective action.
- Identify systematic problems before they become serious.

- Identify and track client complaints and provide resolution.

**12.2.1 Corrective Action Report (CAR)** - is used to document the following types of corrective actions:

- Client complaints
- Deviations from an established procedure or SOP
- QC outside of limits
- Isolated reporting / calculation errors
- Reissued reports

This will provide background documentation to enable root cause analysis and preventive action.

**12.2.2 Nonconformance Database** - is used to document the following types of corrective actions:

- Internal Audits findings
- External Audit findings
- Questionable trends that are found in the review of CARs
- Issues found while reviewing CARs that warrant further investigation.
- Corrective actions that cross multiple departments in the laboratory.
- Systematic reporting / calculation errors
- Identified poor process or method performance trends
- Data recall
- Corrective actions that cross multiple departments in the laboratory
- Failed or unacceptable PT results
- Excessive revised reports

This too will provide background documentation to enable root cause analysis and preventive action.

### **12.3 Closed Loop Corrective Action Process**

Any employee in the company can initiate a corrective action. There are four main components to a closed-loop corrective action process once an issue has been identified: Cause Analysis, Selection and Implementation of Corrective Actions (both short and long term), Monitoring of the Corrective Actions, and Follow-up.

### **12.3.1 Cause Analysis**

- Upon discovery of a non-conformance event, the event must be defined and documented. A CAR must be initiated, someone is assigned to investigate the issue and the event is investigated for cause. Table 12-1 provides some general guidelines on determining responsibility for assessment.
- The cause analysis step is the key to the process as a long term corrective action cannot be determined until the cause is determined.
- If the cause is not readily obvious, the Technical/Department Manager, Laboratory Director or QA Manager (or QA designee) is consulted.

### **12.3.2 Selection and Implementation of Corrective Actions**

- Where corrective action is needed, the laboratory shall identify potential corrective actions. The action(s) most likely to eliminate the problem and prevent recurrence are selected and implemented. Responsibility for implementation is assigned.
- Corrective actions shall be to a degree appropriate to the magnitude of the problem identified through the cause analysis.
- Whatever corrective action is determined to be appropriate, the laboratory shall document and implement the changes. The CAR is used for this documentation.

### **12.3.3 Root Cause Analysis**

Root Cause Analysis is a class of problem solving (investigative) methods aimed at identifying the basic or causal factor(s) that underlie variation in performance or the occurrence of a significant failure. The root cause may be buried under seemingly innocuous events, many steps preceding the perceived failure. At first glance, the immediate response is typically directed at a symptom and not the cause. Typically, root cause analysis would be best with three or more incidents to triangulate a weakness.

Systematically analyze and document the Root Causes of the more significant problems that are reported. Identify, track, and implement the corrective actions required to reduce the likelihood of recurrence of significant incidents. Trend the Root Cause data from these incidents to identify Root Causes that, when corrected, can lead to dramatic improvements in performance by eliminating entire classes of problems.

Identify the one event associated with problem and ask why this event occurred. Brainstorm the root causes of failures; for example, by asking why events occurred or conditions existed; and then why the cause occurred 5 consecutive times until you get to the root cause. For each of these sub events or causes, ask why it occurred. Repeat the process for the other events associated with the incident.

Root cause analysis does not mean the investigation is over. Look at technique, or other systems outside the normal indicators. Often creative thinking will find root causes that ordinarily would be missed, and continue to plague the laboratory or operation.

#### **12.3.4 Monitoring of the Corrective Actions**

- The Laboratory Director, Department Manager and/or QA Manager are responsible to ensure that the corrective action taken was effective.
- Ineffective actions are documented and re-evaluated until acceptable resolution is achieved. Department Managers are accountable to the Laboratory Director to ensure final acceptable resolution is achieved and documented appropriately.
- The QA Manager reviews CARs monthly for trends. Highlights are included in the QA monthly report (refer to Section 16). If a significant trend develops that adversely affects quality, an audit of the area is performed and corrective action implemented.
- Any out-of-control situations that are not addressed acceptably at the laboratory level may be reported to the Corporate Quality Director by the QA Manager, indicating the nature of the out-of-control situation and problems encountered in solving the situation.

#### **12.3.5 Follow-up Audits**

- Follow-up audits may be initiated by the QA Manager and shall be performed as soon as possible when the identification of a nonconformance casts doubt on the laboratory's compliance with its own policies and procedures, or on its compliance with state or federal requirements.
- These audits often follow the implementation of the corrective actions to verify effectiveness. An additional audit would only be necessary when a critical issue or risk to business is discovered.

(Also refer to Section 15.1.4, Special Audits.)

#### **12.4 Technical Corrective Actions**

In addition to providing acceptance criteria and specific protocols for technical corrective actions in the method SOPs, the laboratory has general procedures to be followed to determine when departures from the documented policies and procedures and quality control have occurred (refer to Section 11). The documentation of these procedures is through the use of a CAR.

Table 12-1 includes examples of general technical corrective actions. For specific criteria and corrective actions, refer to the analytical methods or specific method SOPs. The laboratory may also maintain Work Instructions on these items that are available upon request.

Table 12-1 provides some general guidelines for identifying the individual(s) responsible for assessing each QC type and initiating corrective action. The table also provides general guidance on how a data set should be treated if associated QC measurements are unacceptable. Specific procedures are included in Method SOPs, Work Instructions and QAM Sections 19 and 20. All corrective actions are reviewed monthly, at a minimum, by the QA Manager and highlights are included in the QA monthly report.

To the extent possible, samples shall be reported only if all quality control measures are acceptable. If the deficiency does not impair the usability of the results, data will be reported with an appropriate data qualifier and/or the deficiency will be noted in the case narrative. Where

sample results may be impaired, the Project Manager is notified by a CAR and appropriate corrective action (e.g., reanalysis) is taken and documented.

## **12.5 Basic Corrections**

When mistakes occur in records, each mistake shall be crossed-out, [not obliterated (e.g. no white-out)], and the correct value entered alongside. All such corrections shall be initialed (or signed) and dated by the person making the correction. In the case of records stored electronically, the original “uncorrected” file must be maintained intact and a second “corrected” file is created.

This same process applies to adding additional information to a record. All additions made later than the initial must also be initialed (or signed) and dated.

When corrections are due to reasons other than obvious transcription errors, the reason for the corrections (or additions) shall also be documented.

Figure 12-1. Example - Corrective Action Report

**Corrective Action Report: TAPLIMS.Phoenix - Mary Tyer**

Corrective Action Supervisor QA PM Print Exit

CAR No. <NEW> Status Open Client Complaint  Commit  
Entered By Mary Tyer Date Entered 11/11/2010 NCR  Cancel

Issue | Batch/Work Order Information | Supervisor | Quality Assurance | Project Management

Issue Information

Employee None Specified Date of Occurrence 11/11/2010 Additional Issue Notes

Department None Specified Instrument

Issue Description

Issue Cause Description

Employee Oversight Description

Internal Corrective Action Description

Start | Element DataSyst... | Corrective Acti... | Inbox - Microsoft ... | 2010 QAM Template | Draft QAM 2010 - ... | Microsoft Office ... | 8:00 AM

Table 12-1. Example – General Corrective Action Procedures

<b>QC Activity (Individual Responsible for Initiation/Assessment)</b>	<b>Acceptance Criteria</b>	<b>Recommended Corrective Action</b>
Initial Instrument Blank  (Analyst)	- Instrument response < MDL.	- Prepare another blank. - If same response, determine cause of contamination: reagents, environment, instrument equipment failure, etc.
Initial Calibration Standards  (Analyst, Technical /Department Manager(s))	- Correlation coefficient > 0.99 or standard concentration value. - % Recovery within acceptance range. - See details in Method SOP.	- Reanalyze standards. - If still unacceptable, remake standards and recalibrate instrument. - Perform Instrument maintenance
Independent Calibration Verification (Second Source)  (Analyst, Technical /Department Manager(s))	- % Recovery within acceptance criteria	- Remake and reanalyze standard. - If still unacceptable, then remake calibration standards or use new primary standards and recalibrate instrument.
Continuing Calibration Standards  (Analyst, Data Reviewer)	- % Recovery within acceptance criteria	- Reanalyze standard. - If still unacceptable, then recalibrate and rerun affected samples.
Reporting Limit Verification Standards  (Analyst, Data Reviewer)	- % Recovery within acceptance criteria	- Reanalyze standard. - If still unacceptable, batch must be re-prepared and re-analyzed.
Duplicate (Analyst, Data Reviewer)	- % RPD within acceptance criteria	- Reanalyze once, evaluate. Flag data if reanalysis remains out of control outside of limit.
Matrix Spike / Matrix Spike Duplicate (MS/MSD)  (Analyst, Data Reviewer)	- % Recovery within acceptance criteria  - % RPD within acceptance criteria	- If the acceptance criteria for duplicates or matrix spikes are not met because of matrix interferences, the acceptance of the analytical batch is determined by the validity of the LCS. - If the LCS is within acceptable limits the batch is acceptable. - The results of the duplicates, matrix spikes and the LCS are reported with the data set. - For matrix spike or duplicate results outside criteria the data for that sample shall be reported with qualifiers.

QC Activity (Individual Responsible for Initiation/Assessment)	Acceptance Criteria	Recommended Corrective Action
Laboratory Control Sample / Laboratory Control Sample Duplicate (LCS/LCSD)  (Analyst, Data Reviewer)	<ul style="list-style-type: none"> <li>- % Recovery within acceptance criteria</li> <li>- % RPD within acceptance criteria</li> </ul>	<ul style="list-style-type: none"> <li>- Batch must be re-prepared and re-analyzed. This includes any allowable marginal exceedance.</li> <li>When not using marginal exceedances, the following exceptions apply:               <ol style="list-style-type: none"> <li>1) when the acceptance criteria for the positive control are exceeded high (i.e., high bias) and there are associated samples that are non-detects, then those non-detects may be reported with data qualifying codes;</li> <li>2) when the acceptance criteria for the positive control are exceeded low (i.e., low bias), those sample results may be reported if they exceed a maximum regulatory limit/decision level with data qualifying codes.</li> </ol> </li> <li><b>Note:</b> If there is insufficient sample or the holding time cannot be met, contact client and report with flags.</li> </ul>
Surrogates  (Analyst, Data Reviewer)	<ul style="list-style-type: none"> <li>- % Recovery within acceptance criteria</li> </ul>	<ul style="list-style-type: none"> <li>- Individual sample must be repeated. Place comment on whether initial results confirmed/did not confirm in LIMS.</li> <li>- Surrogate results outside criteria shall be reported with qualifiers.</li> </ul>
Internal Standards  (Analyst, Data Reviewer)	Refer to Method SOP.	<ul style="list-style-type: none"> <li>- Evaluate data and instrument. If no instrument issue found, flag data.</li> </ul>
Method Blank (MB)  (Analyst, Data Reviewer)	< Reporting Limit <sup>1</sup>	<ul style="list-style-type: none"> <li>- Reanalyze blank.</li> <li>- If still positive, determine source of contamination. If necessary, reprocess (i.e. digest or extract) entire sample batch. Report blank results.</li> <li>- Qualify the result(s) if the concentration of a targeted analyte in the MB is at or above the reporting limit AND is &gt; 1/10 of the amount measured in the sample.</li> </ul>
Proficiency Testing (PT) Samples  (QA Manager, Technical / Department Manager(s))	<ul style="list-style-type: none"> <li>- Criteria supplied by PT Supplier.</li> </ul>	<ul style="list-style-type: none"> <li>- Any failures or warnings must be investigated for cause. Failures may result in the need to repeat a PT sample to show the problem is corrected.</li> </ul>

<b>QC Activity (Individual Responsible for Initiation/Assessment)</b>	<b>Acceptance Criteria</b>	<b>Recommended Corrective Action</b>
Internal / External Audits  (QA Manager, Technical /Department Manager(s) Laboratory Director)	- Defined in Quality System documentation such as SOPs, QAM, etc..	- Non-conformances must be investigated and necessary corrections must be made.
Reporting / Calculation Errors  (Depends on issue – possible individuals include: Analysts, Data Reviewers, Project Managers, Technical/ Department Managers, QA Manager, Corporate QA, Corporate Management)	- SOP CW-L-S-002, Internal Investigation of Potential Data Discrepancies and Determination for Data Recall.	- Corrective action is determined by type of error. Follow the procedures in SOP CW-L-S-002 and your lab's CA SOP.
Client Complaints  (Project Managers, Lab Director/Manager, Sales and Marketing)		- Corrective action is determined by the type of complaint. For example, a complaint regarding an incorrect address on a report will result in the report being corrected and then follow-up must be performed on the reasons the address was incorrect (e.g., database needs to be updated).
QA Monthly Report  (QA Manager, Lab Director/Manager, Technical / Department Manager)	- QAM, SOPs.	- Corrective action is determined by the type of issue. For example, CARs for the month are reviewed and possible trends are investigated.
Health and Safety Violation  (Safety Officer, Lab Director, Technical /Department Manager(s))	- Environmental Health and Safety (EHS) Manual.	- Non-conformance is investigated and corrected immediately or reported via the KMI Incident Tracking Online Program.

**Note:**

1. These tables provide general corrective action procedures. Not all QC samples are listed in the table. Frequency, acceptance criteria and corrective actions may vary. Standard Operating Procedures (SOPs) provide detailed information on corrective action for each method or procedure.
2. For instrument and analytical QC, the acceptance criteria are that listed in the applicable method or program (e.g. AIHA) requirements). If no criteria are listed the laboratory utilizes Arizona Department of Health Services default limits or generates control limits using historical laboratory data.
3. QC acceptance criteria is listed in SOPs. QC limits can be found in LIMS.

**SECTION 13. PREVENTIVE ACTION / IMPROVEMENT****13.1 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 and continuous process of improvement activities that can be initiated through feedback from clients, employees, business providers, and affiliates. The QA Department has the overall responsibility to ensure that the preventive action process is in place, and that relevant information on actions is submitted for management review.

Dedicating resources to an effective preventive action system emphasizes the laboratory's commitment to its Quality Program. It is beneficial to identify and address negative trends before they develop into complaints, problems and corrective actions. Additionally, customer service and client satisfaction can be improved through continuous improvements to laboratory systems.

Opportunities for improvement may be discovered during management reviews, the monthly QA Metrics Report, evaluation of internal or external audits, results & evaluation of proficiency testing (PT) performance, data analysis & review processing operations, client complaints, staff observation, etc.

The monthly Management Systems Metrics Report shows performance indicators in all areas of the laboratory and 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 in evaluating the management and quality system performance on an ongoing basis and provide a tool for identifying areas for improvement.

The laboratory's corrective action process is integral to implementation of preventive actions. A critical piece of the corrective action process is the implementation of actions to prevent further occurrence of a non-compliance event. Historical review of corrective action provides a valuable mechanism for identifying preventive action opportunities.

**13.1.1** The following elements are part of a preventive action system:

- 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 and management review.

**13.1.2** Any Preventive Actions undertaken or attempted shall be taken into account during the annual Management Systems Review (Section 16). A highly detailed report is not required; however, a summary of successes and failures within the preventive action program is sufficient to provide management with a measurement for evaluation.

## **SECTION 14. CONTROL OF RECORDS**

The laboratory maintains a records management system appropriate to its needs and that complies with applicable standards or regulations as required. The system produces unequivocal, accurate records that document all laboratory activities. The laboratory retains all original observations, calculations and derived data, calibration records and a copy of the analytical report for a minimum of five years after it has been issued.

### **14.1 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 14-1. Quality records are maintained by the QA Department which is backed up as part of the regular laboratory backup. Records are of two types; either electronic or hard copy paper formats depending on whether the record is computer or hand generated (some records may be in both formats). Technical records are maintained by the QA Department and Corporate IT Department.

**Table 14-1. Record Index<sup>1</sup>**

	<b><u>Record Types</u><sup>1</sup>:</b>	<b><u>Retention Time:</u></b>
<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>- MDLs/IDLs/DOCs</li> <li>- Lab Reports</li> </ul>	5 Years from analytical report issue*

	<b>Record Types <sup>1</sup>:</b>	<b>Retention Time:</b>
<b>Official Documents</b>	- Quality Assurance Manual (QAM) - Work Instructions - Policies - SOPs - Policy Memorandums - Manuals	5 Years from document retirement date*
<b>QA Records</b>	- Internal & External Audits/Responses - Certifications - Corrective/Preventive Actions - Management Reviews - Method & Software Validation / Verification Data - Data Investigation	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>	- Sample Receipt & COC Documentation - Contracts and Amendments - Correspondence - QAPP - SAP - Telephone Logbooks - Lab Reports	5 Years from analytical report issue*
<b>Administrative Records</b>	Finance and Accounting	10 years
	EH&S Manual, Permits	7 years
	Disposal Records	Indefinitely
	Employee Handbook	Indefinitely
	Personnel files, Employee Signature & Initials, Administrative Training Records (e.g., Ethics)	7 Years (HR Personnel Files must be maintained indefinitely)
	Administrative Policies Technical Training Records	7 years

<sup>1</sup> Record Types encompass hardcopy and electronic records.

<sup>2</sup> Examples of Logbook types: Maintenance, Instrument Run, Preparation (standard and samples), Standard and Reagent Receipt, Archiving, Balance Calibration, Temperature (hardcopy or electronic records).

\* Exceptions listed in Table 14-2.

**14.1.1** All records are stored and retained in such a way that they are secure and readily retrievable at the laboratory facility that provides a suitable environment to prevent damage or deterioration and to prevent loss. All records shall be protected against fire, theft, loss, environmental deterioration, and vermin. In the case of electronic records, electronic or magnetic sources, storage media are protected from deterioration caused by magnetic fields and/or electronic deterioration.

Access to the data is limited to laboratory and company employees and shall be documented with an access log. If records are archived off-site they are stored in a secure location where a record is maintained of any entry into the storage facility. Whether on-site or off-site storage is used, logs are maintained in each storage area to note removal and return of records. Retention of records must be maintained on-site at the laboratory for approximately 2 years

after their generation. After two years they may be moved offsite for the remainder of the required storage time. Records are maintained for a minimum of five years unless otherwise specified by a client or regulatory requirement.

For raw data and project records, record retention shall be calculated from the date the project report is issued. For other records, such as Controlled Documents, QA, or Administrative Records, the retention time is calculated from the date the record is formally retired. Records related to the programs listed in Table 14-2 have lengthier retention requirements and are subject to the requirements in Section 14.1.3.

#### **14.1.2 Programs with Longer Retention Requirements**

Some regulatory programs have longer record retention requirements than the standard record retention time. These are detailed in Table 14-2 with their retention requirements. In these cases, the longer retention requirement is enacted. If special instructions exist such that client data cannot be destroyed prior to notification of the client, the container or box containing that data is marked as to who to contact for authorization prior to destroying the data.

**Table 14-2. Example: Special Record Retention Requirements**

<b>Program</b>	<b><sup>1</sup>Retention Requirement</b>
Drinking Water – All States	5 years (project records) 10 years - Radiochemistry (project records)
Drinking Water Lead and Copper Rule	12 years (project records)
Housing and Urban Development (HUD) Environmental Lead Testing	10 years
TSCA - 40 CFR Part 792	10 years after publication of final test rule or negotiated test agreement

<sup>1</sup>Note: Extended retention requirements must be noted with the archive documents or addressed in facility-specific records retention procedures.

**14.1.3** The laboratory has procedures to protect and back-up records stored electronically and to prevent unauthorized access to or amendment of these records. All analytical data is maintained as hard copy or in a secure readable electronic format. For analytical reports that are maintained as copies in PDF format, refer to Section 19.14.1 for more information. See SOPs PE-ADM-002 Data Back-up Procedures and PE-QAD-017 Record Archiving.

**14.1.4** The record keeping system allows for historical reconstruction of all laboratory activities that produced the analytical data, as well as rapid recovery of historical data. The history of the sample from when the laboratory took possession of the samples must be readily understood through the documentation. This shall include inter-laboratory transfers of samples and/or extracts.

- The records include the identity of personnel involved in sampling, sample receipt, preparation, or testing. All analytical work contains the initials (at least) of the personnel involved. The laboratory's copy of the COC is stored with the invoice and the work order sheet generated by the LIMS. The chain of custody would indicate the name of the sampler.

If any sampling notes are provided with a work order, they are kept with this package.

- All information relating to the laboratory facilities equipment, analytical test methods, and related laboratory activities, such as sample receipt, sample preparation, or data verification are documented.
- The record keeping system facilitates the retrieval of all working files and archived records for inspection and verification purposes (e.g., set format for naming electronic files, set format for what is included with a given analytical data set as defined by method SOPs). 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 log or instrument sequence is stored with the data to aid in reconstructing an analytical sequence. Where an analysis is performed without an instrument, bound logbooks or bench sheets are used to record and file data. Standard and reagent information is recorded in logbooks or entered into the LIMS for each method as required.
- Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.
- The reason for a signature or initials on a document is clearly indicated in the records such as "sampled by," "prepared by," "reviewed by", or "analyzed by".
- All generated data except those that are generated by automated data collection systems, are recorded directly, promptly and legibly in permanent dark ink.
- Hard copy data may be scanned into PDF format for record storage as long as the scanning process can be verified in order to ensure that no data is lost and the data files and storage media must be tested to verify the laboratory's ability to retrieve the information prior to the destruction of the hard copy that was scanned. The procedure for this verification can be found in SOP PE-PMD-001 Data Reporting, Validation and Distribution.
- Also refer to Section 19.14.1 'Computer and Electronic Data Related Requirements'.

## **14.2 Technical and Analytical Records**

**14.2.1** The laboratory retains records of original observations, derived data and sufficient information to establish an audit trail, calibration records, staff records and a copy of each analytical report issued, for a minimum of five years unless otherwise specified by a client or regulatory requirement. The records for each analysis shall contain sufficient information to enable the analysis to be repeated under conditions as close as possible to the original. The records shall include the identity of laboratory personnel responsible for sampling, performance of each analysis and reviewing results.

**14.2.2** Observations, data and calculations are recorded real-time and are identifiable to the specific task.

**14.2.3** Changes to hardcopy records shall follow the procedures outlined in Section 12 and 19. Changes to electronic records in LIMS or instrument data are recorded in audit trails.

The essential information to be associated with analysis, such as strip charts, tabular printouts, computer data files, analytical notebooks, and run logs, include:

- laboratory sample ID code;
- Date of analysis; time of analysis is also required if the holding time is seventy-two (72) hours or less, or when time critical steps are included in the analysis (e.g., drying times, incubations, etc.); instrumental analyses have the date and time of analysis recorded as part of their general operations. Where a time critical step exists in an analysis, location for such a time is included as part of the documentation in a specific logbook or on a bench sheet.
- Instrumentation identification and instrument operating conditions/parameters. Operating conditions/parameters are typically recorded in instrument maintenance logs where available or indicated in method SOPs.
- analysis type;
- all manual calculations and manual integrations;
- analyst's or operator's initials/signature;
- Sample preparation including cleanup, separation protocols, incubation periods or subculture, ID codes, volumes, weights, instrument printouts, meter readings, calculations, reagents;
- test results;
- standard and reagent origin, receipt, preparation, and use;
- calibration criteria, frequency and acceptance criteria;
- data and statistical calculations, review, confirmation, interpretation, assessment and reporting conventions;
- quality control protocols and assessment;
- electronic data security, software documentation and verification, software and hardware audits, backups, and records of any changes to automated data entries; and
- Method performance criteria including expected quality control requirements. These are indicated both in the LIMS and on specific analytical report formats.

### **14.3 Laboratory Support Activities**

In addition to documenting all the above-mentioned activities, the following are retained QA records and project records (previous discussions in this section relate where and how these data are stored):

- all original raw data, whether hard copy or electronic, for calibrations, samples and quality control measures, including analysts' work sheets and data output records (chromatograms, strip charts, and other instrument response readout records);
- a written description or reference to the specific test method used which includes a description of the specific computational steps used to translate parametric observations into a reportable analytical value;
- copies of final reports;

- archived SOPs;
- correspondence relating to laboratory activities for a specific project;
- all corrective action reports, audits and audit responses;
- proficiency test results and raw data; and
- results of data review, verification, and crosschecking procedures

#### **14.3.1 Sample Handling Records**

Records of all procedures to which a sample is subjected while in the possession of the laboratory are maintained. These include but are not limited to records pertaining to:

- sample preservation including appropriateness of sample container and compliance with holding time requirement;
- sample identification, receipt, acceptance or rejection and login;
- sample storage and tracking including shipping receipts, sample transmittal / COC forms; and
- procedures for the receipt and retention of samples, including all provisions necessary to protect the integrity of samples.

#### **14.4 Administrative Records**

The laboratory also maintains the administrative records in either electronic or hard copy form. Refer to Table 14-1.

#### **14.5 Records Management, Storage and Disposal**

All records (including those pertaining to test equipment), certificates and reports are safely stored, held secure and in confidence to the client. Certification related records are available upon request.

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.

Records that are stored or generated by computers or personal computers have hard copy, write-protected backup copies, or an electronic audit trail controlling access.

The laboratory has a record management system (a.k.a. document control) for control of laboratory notebooks, instrument logbooks, standards logbooks, and records for data reduction, validation, storage and reporting. Laboratory notebooks are issued on a per analysis basis, and are numbered sequentially. All data are recorded sequentially within a series of sequential notebooks. Bench sheets are filed sequentially. Standards are maintained in the LIMS – no logbooks are used to record that data. Records are considered archived when moved from current storage within the laboratory department.

#### **14.5.1 Transfer of Ownership**

In the event that the laboratory transfers ownership or goes out of business, the laboratory shall ensure that the records are maintained or transferred according to client's instructions. Upon ownership transfer, record retention requirements shall be addressed in the ownership transfer agreement and the responsibility for maintaining archives is clearly established. In addition, in cases of bankruptcy, appropriate regulatory and state legal requirements concerning laboratory records must be followed. In the event of the closure of the laboratory, all records will revert to the control of the corporate headquarters. Should the entire company cease to exist, as much notice as possible will be given to clients and the accrediting bodies who have worked with the laboratory during the previous 5 years of such action.

#### **14.5.2 Records Disposal**

Records are removed from the archive and destroyed after 5 years unless otherwise specified by a client or regulatory requirement. On a project specific or program basis, clients may need to be notified prior to record destruction. Records are destroyed in a manner that ensures their confidentiality such as shredding, mutilation or incineration. (Refer to Tables 14-1 and 14-2).

Electronic copies of records must be destroyed by erasure or physically damaging off-line storage media so no records can be read.

If a third party records management company is hired to dispose of records, a "Certificate of Destruction" is required.

### **SECTION 15. AUDITS**

#### **15.1 Internal Audits**

Internal audits are performed to verify that laboratory operations comply with the requirements of the lab's quality system and with the external quality programs under which the laboratory operates. Audits are planned and organized by the QA staff. Personnel conducting the audits should be independent of the area being evaluated. Auditors will have sufficient authority, access to work areas, and organizational freedom necessary to observe all activities affecting quality and to report the assessments to laboratory management and, when requested, to corporate management.

Audits are conducted and documented as described in the TestAmerica Corporate SOP on performing Internal Auditing, SOP No. CA-Q-S-004. The types and frequency of routine internal audits are described in Table 15-1. Special or ad hoc assessments may be conducted as needed under the direction of the QA staff.

**Table 15-1. Types of Internal Audits and Frequency**

Description	Performed by	Frequency
Quality Systems Audits	QA Department, QA approved designee, or Corporate QA	All areas of the laboratory annually
Method Audits	Joint responsibility: a) QA Manager or designee b) Technical Manager or Designee (Refer to CA-Q-S-004)	Methods Audits Frequency: 50% of methods annually
Special	QA Department or Designee	Surveillance or spot checks performed as needed, e.g., to confirm corrective actions from other audits.
Performance Testing	Analysts with QA oversight	Two successful per year for each TNI 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, TestAmerica's Data Integrity and Ethics Policies, TNI 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 for effectiveness & sustainability. The audit is divided into sections for each operating or support area of the lab, and each section is comprehensive for a given area. The area audits may be performed 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, electronic audit miner programs (e.g., MintMiner) are 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/Department Manager or qualified designee at least every two years. It is also recommended that 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**

The laboratory participates annually or semi-annually in performance audits conducted through the analysis of PT samples provided by a third party. The laboratory generally participates in the following types of PT studies: Water Supply, Water Pollution, Underground Storage Tank, Hazardous Waste, Air, AIHA IHPAT, WASP and other Round Robin studies.

It is TestAmerica's policy that PT samples be treated as typical samples in the production process. Furthermore, where PT samples present special or unique problems, in the regular production process they may need to be treated differently, as would any special or unique request submitted by any client. The QA Manager must be consulted and in agreement with any decisions made to treat a PT sample differently due to some special circumstance.

Written responses to unacceptable PT results are required. In some cases it may be necessary for blind QC samples to be submitted to the laboratory to show a return to control.

#### **15.2 External Audits**

External audits are performed when certifying agencies or clients conduct on-site inspections or submit performance testing samples for analysis. It is TestAmerica's policy to cooperate fully with regulatory authorities and clients. The laboratory makes every effort to provide the auditors with access to personnel, documentation, and assistance. Laboratory supervisors are responsible for providing corrective actions to the QA Manager who coordinates the response for any deficiencies discovered during an external audit. Audit responses are due in the time allotted by the client or agency performing the audit. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

The laboratory cooperates with clients and their representatives to monitor the laboratory's performance in relation to work performed for the client. The client may only view data and systems related directly to the client's work. All efforts are made to keep other client information confidential.

##### **15.2.1 Confidential Business Information (CBI) Considerations**

During on-site audits, auditors may come into possession of information claimed as business confidential. A business confidentiality claim is defined as "a claim or allegation that business information is entitled to confidential treatment for reasons of business confidentiality or a request for a determination that such information is entitled to such treatment." When information is claimed as business confidential, the laboratory must place on (or attach to) the information at the time it is submitted to the auditor, a cover sheet, stamped or typed legend or other suitable form of notice, employing language such as "trade secret", "proprietary" or

“company confidential”. Confidential portions of documents otherwise non-confidential must be clearly identified. CBI may be purged of references to client identity by the responsible laboratory official at the time of removal from the laboratory. However, sample identifiers may not be obscured from the information. Additional information regarding CBI can be found in within the 2009 TNI standards.

### **15.3 Audit Findings**

Audit findings are documented using the corrective action process and Non Conformance database. The laboratory’s corrective action responses for both types of audits may include action plans that could not be completed within a predefined timeframe. In these instances, a completion date must be set and agreed to by operations management and the QA Manager.

Developing and implementing corrective actions to findings is the responsibility of the Technical/Department Manager where the finding originated. Findings that are not corrected by specified due dates are reported monthly to management in the QA monthly report. When requested, a copy of the audit report and the labs corrective action plan will be forwarded to Corporate Quality.

If any audit finding casts doubt on the effectiveness of the operations or on the correctness or validity of the laboratory’s test results, the laboratory shall take timely corrective action, and shall notify clients in writing if the investigations show that the laboratory results have been affected. Once corrective action is implemented, a follow-up audit is scheduled to ensure that the problem has been corrected.

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.

## **SECTION 16. MANAGEMENT REVIEWS**

### **16.1 Quality Assurance Report**

A comprehensive QA Report shall be prepared each month by the laboratory’s QA Department and forwarded to the Laboratory Director, Technical Managers or designee, their Quality Director as well as the General Manager. All aspects of the QA system are reviewed to evaluate the suitability of policies and procedures. During the course of the year, the Laboratory Director, General Manager or Corporate QA may request that additional information be added to the report.

On a monthly basis, Corporate QA compiles information from all the monthly laboratory reports. The Corporate Quality Directors prepare a report that includes a compilation of all metrics and notable information and concerns regarding the QA programs within the laboratories. The report also includes a listing of new regulations that may potentially impact the laboratories. This report is presented to the Executive Committee and General Managers.

## **16.2 Annual Management Review**

The senior lab management team (Laboratory Director, Technical Managers or designee, QA Manager) conducts a review annually of its quality systems and LIMS to ensure its continuing suitability and effectiveness in meeting client and regulatory requirements and to introduce any necessary changes or improvements. It will also provide a platform for defining goals, objectives and action items that feed into the laboratory planning system. Corporate Operations and Corporate QA personnel can be included in this meeting at the discretion of the Laboratory Director. The LIMS review consists of examining any audits, complaints or concerns that have been raised through the year that are related to the LIMS. The laboratory will summarize any critical findings that can not be solved by the lab and report them to Corporate IT.

This management systems review (Corporate SOP No. CA-Q-S-008 & Work Instruction No. CA-Q-WI-020) uses information generated during the preceding year to assess the “big picture” by ensuring that routine actions taken and reviewed on a monthly basis are not components of larger systematic concerns. The monthly review should keep the quality systems current and effective, therefore, the annual review is a formal senior management process to review specific existing documentation. Significant issues from the following documentation are compiled or summarized by the QA Manager prior to the review meeting:

- Matters arising from the previous annual review.
- Prior Monthly QA Reports issues.
- Laboratory QA Metrics.
- Review of report reissue requests.
- Review of client feedback and complaints.
- Issues arising from any prior management or staff meetings.
- Minutes from prior senior lab management meetings. Issues that may be raised from these meetings include:
  - Adequacy of staff, equipment and facility resources.
  - Adequacy of policies and procedures.
  - Future plans for resources and testing capability and capacity.
- The annual internal double blind PT program sample performance (if performed),
- Compliance to the Ethics Policy and Data Integrity Plan. Including any evidence/incidents of inappropriate actions or vulnerabilities related to data Integrity.

A report is generated by the QA Manager and management. The report is distributed to the appropriate General Manager and the Quality Director. The report includes, but is not limited to:

- The date of the review and the names and titles of participants.
- A reference to the existing data quality related documents and topics that were reviewed.
- Quality system or operational changes or improvements that will be made as a result of the review [e.g., an implementation schedule including assigned responsibilities for the changes (Action Table)].

Changes to the quality systems requiring update to the laboratory QA Manual shall be included in the next revision of the QA Manual.

### **16.3 Potential Integrity Related Managerial Reviews**

Potential integrity issues (data or business related) must be handled and reviewed in a confidential manner until such time as a follow-up evaluation, full investigation, or other appropriate actions have been completed and issues clarified. TestAmerica's Corporate Data Investigation/Recall SOP shall be followed (SOP No. CW-L-S-002). All investigations that result in finding of inappropriate activity are documented and include any disciplinary actions involved, corrective actions taken, and all appropriate notifications of clients.

TestAmerica's CEO, VP of Quality, Technical & Operations Support, General Managers and Quality Directors receive a monthly report from the Corporate Quality Director summarizing any current data integrity or data recall investigations. The General Manager's are also made aware of progress on these issues for their specific labs.

## **SECTION 17. PERSONNEL**

### **17.1 Overview**

The laboratory's management believes that its highly qualified and professional staff is the single most important aspect in assuring a high level of data quality and service. The staff consists of professionals and support personnel as outlined in the organization chart in Figure 4-1.

All personnel must demonstrate competence in the areas where they have responsibility. Any staff that is undergoing training shall have appropriate supervision until they have demonstrated their ability to perform their job function on their own. Staff shall be qualified for their tasks based on appropriate education, training, experience and/or demonstrated skills as required.

The laboratory employs sufficient personnel with the necessary education, training, technical knowledge and experience for their assigned responsibilities.

All personnel are responsible for complying with all QA/QC requirements that pertain to the laboratory and their area of responsibility. Each staff member must have a combination of experience and education to adequately demonstrate a specific knowledge of their particular area of responsibility. Technical staff must also have a general knowledge of lab operations, test methods, QA/QC procedures and records management.

Laboratory management is responsible for formulating goals for lab staff with respect to education, training and skills and ensuring that the laboratory has a policy and procedures for identifying training needs and providing training of personnel. The training shall be relevant to the present and anticipated responsibilities of the lab staff.

The laboratory only uses personnel that are employed by or under contract to, the laboratory. Contracted personnel, when used, must meet competency standards of the laboratory and work in accordance to the laboratory's quality system.

## 17.2 Education and Experience Requirements for Technical Personnel

The laboratory makes every effort to hire analytical staffs that possess a college degree (AA, BA, BS) in an applied science with some chemistry in the curriculum. Exceptions can be made based upon the individual's experience and ability to learn. 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 site's Human Resources web-page (Also see Section 4 of this manual 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:

**Table 17-2. General Personnel Educational Requirements/Experience**

Specialty	Education	Experience
Extractions, Digestions, some electrode methods (pH, DO, 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

Specialty	Education	Experience
Technical/Department Managers – <b>General</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/Department Managers – <b>Wet Chem</b> only (no advanced instrumentation)	Associates degree in an applied science or engineering or 2 years of college with 16 semester hours in chemistry	And 2 years relevant experience
Technical/Department Managers - 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

Table 17-2. Personnel Educational Requirements/Experience: Industrial Hygiene

Specialty	Education	Experience
Technician - Industrial Hygiene	H.S. Diploma or equivalent	On the job training (OJT)  Demonstrated and documented ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples (IDOCs). This demonstration shall be done at a minimum of every six (6) months and documented.

Specialty	Education	Experience
Analyst - Industrial Hygiene	Bachelors Degree in Chemistry or related science	One year or more prior analytical laboratory experience desired.  Demonstrated and documented ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples (IDOCs). This demonstration shall be done at a minimum of every six (6) months and documented.
Technical Director – Industrial Hygiene  * The TD shall be present on site at least 20 hours per week or 50 percent of the laboratory operating hours (whichever is less) to address technical issues for laboratory staff and customers.	Bachelors Degree in an applicable physical or biological science  An advanced (MS, PhD.) degree may substitute for one year of experience	And a minimum of 3 years relevant nonacademic analytical chemistry experience which includes a minimum of 2 years industrial hygiene experience within the scope of accreditation

When an analyst does not meet these requirements, they can perform a task under the direct supervision of a qualified analyst, peer reviewer or Technical/Department Managers, and are considered an analyst in training. The person supervising an analyst in training is accountable for the quality of the analytical data and must review and approve data and associated corrective actions.

### 17.3 Training

The laboratory is committed to furthering the professional and technical development of employees at all levels.

Orientation to the laboratory's policies and procedures, in-house method training, and employee attendance at outside training courses and conferences all contribute toward employee proficiency. Examples of various areas of required employee training are listed in Table 17-3.

**Table 17-3. Required Employee Training**

Required Training	Time Frame	Employee Type
Environmental Health & Safety	Prior to lab work	All
Ethics – New Hires	1 week of hire	All
Ethics – Comprehensive	90 days of hire	All
Data Integrity	30 days of hire	Technical and PMs
Quality Assurance	90 days of hire	All

Required Training	Time Frame	Employee Type
Ethics – Refresher	Annually (Training sessions presented throughout the year)	All
Initial Demonstration of Capability (IDOC)	Prior to unsupervised method performance	Technical
Complete a training course (an in-house course is acceptable) for the applicable analysis. Courses on sample preparation and instrument analysis may be taken separately or combined.	Prior to performing unsupervised analysis on laboratory samples.	Industrial Hygiene Technician/Analyst
Minimum of twenty (20) business days of hands-on experience conducting analyses in an industrial hygiene laboratory	Before initiation of independent work on customer samples.	Industrial Hygiene Technician/Analyst

The laboratory maintains records of relevant authorization/competence, education, professional qualifications, training, skills and experience of technical personnel (including contracted personnel) as well as the date that approval/authorization was given. These records are kept on file at the laboratory. Also refer to “Demonstration of Capability” in Section 19.

The training of technical staff is kept up to date by:

- Each employee must have documentation in their training file that they have read, understood and agreed to follow the most recent version of the laboratory QA Manual and SOPs in their area of responsibility. This documentation is updated as SOPs are updated.
- Documentation from any training courses or workshops on specific equipment, analytical techniques or other relevant topics are maintained in their training file.
- Documentation of proficiency (refer to Section 19).
- An Ethics Agreement signed by each staff member (renewed each year) and evidence of annual ethics training.
- A Confidentiality Agreement signed by each staff member signed at the time of employment.
- Human Resources maintains documentation and attestation forms on employment status & records; benefit programs; timekeeping/payroll; and employee conduct (e.g., ethics). This information is maintained in the employee’s secured personnel file.

Further details of the laboratory's training program are described in the SOP PE-QAD-008 Personnel Certification and Training.

#### **17.4 Data Integrity and Ethics Training Program**

Establishing and maintaining a high ethical standard is an important element of a Quality System. Ethics and data integrity training is integral to the success of TestAmerica and is provided for each employee at TestAmerica. It is a formal part of the initial employee orientation

within 1 week of hire followed by technical data integrity training within 30 days, comprehensive training within 90 days, and an annual refresher for all employees. Senior management at each facility performs the ethics training for their staff.

In order to ensure that all personnel understand the importance TestAmerica places on maintaining high ethical standards at all times; TestAmerica has established a Corporate Ethics Policy (Policy No. CW-L-P-004) and an Ethics Statement. All initial and annual training is documented by signature on the signed Ethics Statement demonstrating that the employee has participated in the training and understands their obligations related to ethical behavior and data integrity.

Violations of this Ethics Policy will not be tolerated. Employees who violate this policy will be subject to disciplinary actions up to and including termination. Criminal violations may also be referred to the Government for prosecution. In addition, such actions could jeopardize TestAmerica's ability to do work on Government contracts, and for that reason, TestAmerica has a Zero Tolerance approach to such violations.

Employees are trained as to the legal and environmental repercussions that result from data misrepresentation. Key topics covered in the presentation include:

- Organizational mission and its relationship to the critical need for honesty and full disclosure in all analytical reporting.
- Ethics Policy
- How and when to report ethical/data integrity issues. Confidential reporting.
- Record keeping.
- Discussion regarding data integrity procedures.
- Specific examples of breaches of ethical behavior (e.g. peak shaving, altering data or computer clocks, improper macros, etc., accepting/offering kickbacks, illegal accounting practices, unfair competition/collusion)
- Internal monitoring. Investigations and data recalls.
- Consequences for infractions including potential for immediate termination, debarment, or criminal prosecution.
- Importance of proper written narration / data qualification by the analyst and project manager with respect to those cases where the data may still be usable but are in one sense or another partially deficient.

Additionally, a data integrity hotline (1-800-736-9407) is maintained by TestAmerica and administered by the Corporate Quality Department.

## **SECTION 18. ACCOMMODATIONS AND ENVIRONMENTAL CONDITIONS**

### **18.1 Overview**

The laboratory is a 24,000 ft<sup>2</sup> secure laboratory facility with controlled access and designed to accommodate an efficient workflow and to provide a safe and comfortable work environment for

employees. All visitors sign in and are escorted by laboratory personnel. Access is controlled by various measures.

The laboratory is equipped with structural safety features. Each employee is familiar with the location, use, and capabilities of general and specialized safety features associated with their workplace. The laboratory provides and requires the use of protective equipment including safety glasses, protective clothing, gloves, etc., OSHA and other regulatory agency guidelines regarding required amounts of bench and fume hood space, lighting, ventilation (temperature and humidity controlled), access, and safety equipment are met or exceeded.

Traffic flow through sample preparation and analysis areas is minimized to reduce the likelihood of contamination. Adequate floor space and bench top area is provided to allow unencumbered sample preparation and analysis space. Sufficient space is also provided for storage of reagents and media, glassware, and portable equipment. Ample space is also provided for refrigerated sample storage before analysis and archival storage of samples after analysis. Laboratory HVAC and deionized water systems are designed to minimize potential trace contaminants.

The laboratory is separated into specific areas for sample receiving, sample preparation, volatile organic sample analysis, non-volatile organic sample analysis, inorganic sample analysis, microbiological sample analysis, and administrative functions.

## **18.2 Environment**

Laboratory accommodation, test areas, energy sources, and 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.

When any of the method or regulatory required environmental conditions change to a point where they may adversely affect test results, analytical testing will be discontinued until the environmental conditions are returned to the required levels.

Environmental conditions of the facility housing the computer network and LIMS are regulated to protect against raw data loss.

## **18.3 Work Areas**

There is effective separation between neighboring areas when the activities therein are incompatible with each other. Examples include:

- Microbiological culture handling and sample incubation areas.

- 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.

Refer to the following documents and procedures for specific requirements for microbiological laboratory facility requirements.

- Standard Methods, 20<sup>th</sup> Ed., 9020B, Sec. 2
- TNI V1M5, 1.7.3.7.a
- EPA Manual for the Certification of Laboratories Analyzing Drinking Water, 5<sup>th</sup> Edition

#### **18.4 Floor Plan**

A floor plan can be found in Appendix 1.

#### **18.5 Building Security**

Magnetic building keys and alarm codes are distributed to employees as necessary. Access to the laboratory is controlled to prevent entry by non-laboratory personnel.

Visitors to the laboratory sign in a visitor's logbook. A visitor is defined as any person who visits the laboratory who is not an employee of the laboratory. In addition to signing into the laboratory, the Environmental, Health and Safety Manual contain requirements for visitors and vendors. There are specific safety forms that must be reviewed and signed.

Visitors (with the exception of company employees) are escorted by laboratory personnel at all times, or the location of the visitor is noted in the visitor's logbook

## SECTION 19. TEST METHODS AND METHOD VALIDATION

### 19.1 Overview

The laboratory uses methods that are appropriate to meet our clients' requirements and that are within the scope of the laboratory's capabilities. These include sampling, handling, transport, storage and preparation of samples, and, where appropriate, an estimation of the measurement of uncertainty as well as statistical techniques for analysis of environmental data.

Instructions are available in the laboratory for the operation of equipment as well as for the handling and preparation of samples. All instructions, Standard Operating Procedures (SOPs), reference methods and manuals relevant to the working of the laboratory are readily available to all staff. Deviations from published methods are documented (with justification) in the laboratory's approved SOPs. SOPs are submitted to clients for review at their request. Significant deviations from published methods require client approval and regulatory approval where applicable.

### 19.2 Standard Operating Procedures (SOPS)

The laboratory maintains SOPs that accurately reflect all phases of the laboratory such as assessing data integrity, corrective actions, handling customer complaints as well as all analytical methods and sampling procedures. The method SOPs are derived from the most recently promulgated/approved, published methods and are specifically adapted to the laboratory facility. Modifications or clarifications to published methods are clearly noted in the SOPs. All SOPs are controlled by the laboratory.

- All SOPs contain a revision number, effective date, and appropriate approval signatures. SOP copies (as uncontrolled documents) are available online to all staff. Controlled copies are utilized as requested by laboratory personnel.
- Procedures for writing a SOP are incorporated by reference to TestAmerica's Corporate SOP CW-Q-S-002 entitled 'Writing a Standard Operating Procedure (SOP)', or the laboratory's SOP PE-QAD-014 Creation and Maintenance of SOPs.
- SOPs are reviewed at a minimum of every 2 years (annually for Drinking Water SOPs), and where necessary, revised to ensure continuing suitability and compliance with applicable requirements.

### 19.3 Laboratory Methods Manual

For each test method, the laboratory shall have available the published referenced method as well as the laboratory developed SOP.

**Note:** If more stringent standards or requirements are included in a mandated test method or regulation than those specified in this manual, the laboratory shall demonstrate that such requirements are met. If it is not clear which requirements are more stringent, the standard from the method or regulation is to be followed. Any exceptions or deviations from the referenced methods or regulations are noted in the specific analytical SOP.

The laboratory maintains an SOP Index for both technical and non-technical SOPs. Technical SOPs are maintained to describe a specific test method. Non-technical SOPs are maintained to describe functions and processes not related to a specific test method.

## 19.4 Selection of Methods

Since numerous methods and analytical techniques are available, continued communication between the client and laboratory is imperative to assure the correct methods are utilized. Once client methodology requirements are established, this and other pertinent information is summarized by the Project Manager. These mechanisms ensure that the proper analytical methods are applied when the samples arrive for log-in. For non-routine analytical services (e.g., special matrices, non-routine compound lists), the method of choice is selected based on client needs and available technology. The methods selected should be capable of measuring the specific parameter of interest, in the concentration range of interest, and with the required precision and accuracy.

### 19.4.1 Sources of Methods

- 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.
- 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)
- Handbook for Analytical Quality Control in Water and Wastewater Laboratories, EPA 600/4-79-019, EPA, March 1979.
- Technical Notes on Drinking Water Methods, EPA-600/R94-173, October 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; Final Update IV, January 2008.
- Annual Book of ASTM Standards, American Society for Testing & Materials (ASTM), Philadelphia, PA.
- 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
- NIOSH Manual of Analytical Methods (NMAM®), 4th ed.
- DHHS (NIOSH) Publication 94-113 (August, 1994), 1st Supplement Publication 96-135, 2nd Supplement Publication 98-119, 3rd Supplement 2003-154 Schlecht, P.C. & O'Connor, P.F. (pfo1@cdc.gov), Eds.

- *Index of Sampling & Analytical Methods, U.S. Department of Labor, Occupational Safety & Health Administration, Revision Date: 21 November 2001.*
- *8015AZ R1, C10 – C32 Hydrocarbons in Soil, Arizona Department of Health Services, Revision 1, September 25<sup>th</sup>, 1998.*
- *VOCs in Vapor by 8260B AZ Method, Arizona Department of Health Services, Revision 0.0, April 4<sup>th</sup> 2009.*
- *Compendium of Methods for the Determination of Toxic Organic Compounds in Ambient Air, EPA-625/R96/010b, January 1999.*
- *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*
- *The Determination of Organo-Phosphorus Pesticides in Municipal and Industrial Wastewater, EPA method 1657.*

The laboratory reviews updated versions to all the aforementioned references for adaptation based upon capabilities, instrumentation, etc., and implements them as appropriate. As such, the laboratory strives to perform only the latest versions of each approved method as regulations allow or require.

Other reference procedures for non-routine analyses may include methods established by specific states (e.g., Underground Storage Tank methods), ASTM or equipment manufacturers. Sample type, source, and the governing regulatory agency requiring the analysis will determine the method utilized.

The laboratory shall inform the client when a method proposed by the client may be inappropriate or out of date. After the client has been informed, and they wish to proceed contrary to the laboratory's recommendation, it will be documented.

#### **19.4.2 Demonstration of Capability**

Before the laboratory may institute a new method and begin reporting results, the laboratory shall confirm that it can properly operate the method. In general, this demonstration does not test the performance of the method in real world samples, but in an applicable and available clean matrix sample. If the method is for the testing of analytes that are not conducive to spiking, demonstration of capability may be performed on quality control samples.

A demonstration of capability (Personnel Training and Certification, PE-QAD-008 and Demonstration of Competency DOC for Industrial Hygiene Fields of Testing Not Covered by AIHA PT Samples, PE-QAD-025) is performed whenever there is a change in instrument type (e.g., new instrumentation), method or personnel (e.g., analyst hasn't performed the test within the last 12 months, in the last 6 months for AIHA test methods).

A method's initial demonstration of capability must be thoroughly documented and approved by the Laboratory Director and Technical/Department Manager along with the QA Manager prior to independently analyzing client samples. All associated documentation must be retained in accordance with the laboratory's archiving procedures.

The laboratory must have an approved SOP, demonstrate satisfactory performance, and conduct an MDL study (when applicable). There may be other requirements as stated within the published method or regulations (i.e., retention time window study).

**Note:** In some instances, a situation may arise where a client requests that an unusual analyte be reported using a method where this analyte is not normally reported. If the analyte is being reported for regulatory purposes, the method must meet all procedures outlined within this QA Manual (SOP, MDL, and Demonstration of Capability). If the client states that the information is not for regulatory purposes, the result may be reported as long as the following criteria are met:

- The instrument is calibrated for the analyte to be reported using the criteria for the method and ICV/CCV criteria are met (unless an ICV/CCV is not required by the method or criteria are per project DQOs).
- The laboratory's nominal or default reporting limit (RL) is equal to the quantitation limit (QL), must be at or above the lowest non-zero standard in the calibration curve and must be reliably determined. Project RLs are client specified reporting levels which may be higher than the QL. Results reported below the QL must be qualified as estimated values. Also see Section 19.6.1.3, Relationship of Limit of Detection (LOD) to Quantitation Limit (QL). The final report must be footnoted: *Reporting Limit based on the low standard of the calibration curve.*
- If applicable, the analyte was be qualified to note that the laboratory is not accredited for the analyte and/or the analyte is not referenced method does not contain the analyte as part of its method compound list.
- The client request is documented and the lab informs the client of its procedure for working with unusual compounds.

#### **19.4.3 Analyst Initial Demonstration of Capability (IDOC) Procedures**

Prior to reporting any data, each analyst must have on file with the QA office information demonstrating proficiency with the analytical technique. Both precision and accuracy are measured for the target analytes.

**19.4.3.1** The spiking standard used must be prepared independently from those used in instrument calibration.

**19.4.3.2** The analyte(s) shall be diluted in a volume of clean matrix sufficient to prepare four aliquots at the concentration specified by a method or the laboratory SOP.

**19.4.3.3** At least four aliquots shall be prepared (including any applicable clean-up procedures) and analyzed according to the test method (either concurrently or over a period of days).

**19.4.3.4** Using all of the results, calculate the mean recovery in the appropriate reporting units and the standard deviations for each parameter of interest.

**19.4.3.5** When it is not possible to determine the mean and standard deviations, such as for presence, absence and logarithmic values, the laboratory will assess performance against criteria described in the Method SOP.

**19.4.3.6** Compare the information obtained above to the corresponding acceptance criteria for precision and accuracy in the test method (if applicable) or in laboratory generated acceptance criteria (LCS or interim criteria) if there is no mandatory criteria established. If any one of the parameters do not meet the acceptance criteria, the performance is unacceptable for that parameter.

**19.4.3.7** When one or more of the tested parameters fail at least one of the acceptance criteria, the analyst must proceed according to either option listed below:

- Locate and correct the source of the problem.
- Beginning with 19.4.3.3 above, repeat the test for all parameters that failed to meet criteria. Repeated failure, however, will confirm a general problem with the measurement system. If this occurs, locate and correct the source of the problem and repeat the test for all compounds of interest beginning with 19.4.3.1 above.

Note: Results of successive LCS analyses can be used to fulfill the DOC requirement.

**19.4.3.8** For AIHA Demonstration of Proficiency, all analysts and technicians shall have demonstrated ability to produce reliable results through accurate analysis of certified reference materials (CRMs), proficiency testing samples, or in-house quality control samples. This demonstration shall be done at a minimum of every six (6) months and documented.

A certification statement (refer to Figure 19-1) shall be used to document the completion of each initial demonstration of capability for all NELAC and AIHA listed methods. A copy of the certification is archived in the analyst's training folder.

Methods on line prior to the effective date of this Section shall be updated to the procedures outlined above as new analysts perform their demonstration of capability. A copy of the new record will replace that which was used for documentation in the past. At a minimum, the precision and accuracy of four mid-level laboratory control samples (LCS) must have been compared to the laboratory's quality control acceptance limits. For AIHA, refer to the individual analytical SOP for precision and accuracy demonstration.

**19.4.3.9** For additional information see the laboratory SOP PE-QAD-015 Initial Demonstration of Capability.

## **19.5 Laboratory Developed Methods and Non-Standard Methods**

Any new method developed by the laboratory must be fully defined in an SOP and validated by qualified personnel with adequate resources to perform the method. Method specifications and the relation to client requirements must be clearly conveyed to the client if the method is a non-standard method (not a published or routinely accepted method). The client must also be in agreement to the use of the non-standard method.

## **19.6 Validation of Methods**

Validation is the confirmation by examination and the provision of objective evidence that the particular requirements for a specific intended use are fulfilled.

All non-standard methods, laboratory designed/developed methods, standard methods used outside of their scope, and major modifications to published methods must be validated to confirm they are fit for their intended use. The validation will be as extensive as necessary to meet the needs of the given application. The results are documented with the validation procedure used and contain a statement as to the fitness for use.

#### **19.6.1 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.

##### **19.6.1.1 Determination of Method Selectivity**

Method selectivity is the demonstrated ability to discriminate the analyte(s) of interest from other compounds in the specific matrix or matrices from other analytes or interference. In some cases to achieve the required selectivity for an analyte, a confirmation analysis is required as part of the method.

##### **19.6.1.2 Determination of Method Sensitivity**

Sensitivity can be both estimated and demonstrated. Whether a study is required to estimate sensitivity depends on the level of method development required when applying a particular measurement system to a specific set of samples. Where estimations and/or demonstrations of sensitivity are required by regulation or client agreement, such as the procedure in 40 CFR Part 136 Appendix B, under the Clean Water Act, these shall be followed.

##### **19.6.1.3 Relationship of Limit of Detection (LOD) to the Quantitation Limit (QL)**

An important characteristic of expression of sensitivity is the difference in the LOD and the QL. The LOD is the minimum level at which the presence of an analyte can be reliably concluded. The QL is the minimum concentration of analyte that can be quantitatively determined with acceptable precision and bias. For most instrumental measurement systems, there is a region where semi-quantitative data is generated around the LOD (both above and below the estimated MDL or LOD) and below the QL. In this region, detection of an analyte may be confirmed but quantification of the analyte is unreliable within the accuracy and precision guidelines of the measurement system. When an analyte is detected below the QL, and the presence of the analyte is confirmed by meeting the qualitative identification criteria for the analyte, the analyte can be reliably reported, but the amount of the analyte can only be estimated. If data is to be reported in this region, it must be done so with a qualification that denotes the semi-quantitative nature of the result.

##### **19.6.1.4 Determination of Interferences**

A determination that the method is free from interferences in a blank matrix is performed.

#### **19.6.1.5 Determination of Range**

Where appropriate to the method, the quantitation range is determined by comparison of the response of an analyte in a curve to established or targeted criteria. Generally the upper quantitation limit is defined by highest acceptable calibration concentration. The lower quantitation limit or QL cannot be lower than the lowest non-zero calibration level, and can be constrained by required levels of bias and precision.

#### **19.6.1.6 Determination of Accuracy and Precision**

Accuracy and precision studies are generally performed using replicate analyses, with a resulting percent recovery and measure of reproducibility (standard deviation, relative standard deviation) calculated and measured against a set of target criteria.

#### **19.6.1.7 Documentation of Method**

The method is formally documented in an SOP. If the method is a minor modification of a standard laboratory method that is already documented in an SOP, an SOP Attachment describing the specific differences in the new method is acceptable in place of a separate SOP.

#### **19.6.1.8 Continued Demonstration of Method Performance**

Continued demonstration of Method Performance is addressed in the SOP. Continued demonstration of method performance is generally accomplished by batch specific QC samples such as LCS, method blanks or PT samples.

### **19.7 Method Detection Limits (MDL) / Limits of Detection (LOD)**

Method detection limits (MDL) are initially determined in accordance with 40 CFR Part 136, Appendix B or alternatively by other technically acceptable practices that have been accepted by regulators. MDL is also sometimes referred to as Limit of Detection (LOD). The MDL theoretically represents the concentration level for each analyte within a method at which the Analyst is 99% confident that the true value is not zero. The MDL is determined for each analyte initially during the method validation process and updated as required in the analytical methods, whenever there is a significant change in the procedure or equipment, or based on project specific requirements (refer to 19.7.10). Generally, the analyst prepares at least seven replicates of a 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. Drinking Water method MDLs must be analyzed over a period of 3 or more days.

Refer to the Corporate SOP CA-Q-S-006 Detection Limits or the laboratory's SOP PE-QAD-019 Determination of Method Detection Limits for details on the laboratory's MDL process.

### **19.8 Instrument Detection Limits (IDL)**

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.

IDLs are calculated to determine an instrument's sensitivity independent of any preparation method. IDLs are calculated either using 7 replicate spike analyses, like a MDL but without sample preparation, or by the analysis of 10 instrument blanks and calculating 3 times the absolute value of the standard deviation.

### **19.9 Verification of Detection and Reporting Limits**

Once an MDL is established, it must be verified, on each instrument, by analyzing a quality control sample (prepared as a sample) at no more than 3 times the calculated MDL for single analyte analyses (e.g. most wet chemistry methods, Atomic Absorption, etc.) and no more than 4 times the calculated MDL for multiple analyte methods (e.g. GC, GCMS, ICP, etc.). The analytes must be qualitatively identified. This verification does not apply to methods that are not readily spiked (e.g. pH, turbidity, etc.) or where the lab does not report to the MDL. If the MDL does not verify, then the lab will either not report to the MDL, redevelop their MDL or use the level where qualitative identification is established. MDLs must be verified at least annually

When the laboratory establishes a quantitation limit, it must be initially verified by the analysis of a low level standard or QC sample at 1 - 2 times the reporting limit and annually thereafter. The annual requirement is waived for methods that have an annually verified MDL. The laboratory will comply with any regulatory requirements. Unless there are requirements to the contrary the acceptance criteria is  $\pm 50\%$ .

### **19.10 Retention Time Windows**

Most organic analyses and some inorganic analyses use chromatography techniques for qualitative and quantitative determinations. For every chromatography analysis or as specified in the reference method, each analyte will have a specific time of elution from the column to the detector. This is known as the analyte retention time. The variance in the expected time of elution is defined as the retention time window. As the key to analyte identification in chromatography, retention time windows must be established on every column for every analyte used for that method. These records are kept with the files associated with an instrument for later quantitation of the analytes. Complete details are available in the laboratory SOPs.

### **19.11 Evaluation of Selectivity**

The laboratory evaluates selectivity by following the checks within the applicable analytical methods, which include mass spectral tuning, second column confirmation, ICP interelement interference checks, chromatography retention time windows, sample blanks, spectrochemical, atomic absorption or fluorescence profiles, co-precipitation evaluations and specific electrode response factors.

## **19.12 Estimation of Uncertainty of Measurement**

**19.12.1** Uncertainty is “a parameter associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand” (as defined by the International Vocabulary of Basic and General Terms in Metrology, ISO Geneva, 1993, ISBN 92-67-10175-1). Knowledge of the uncertainty of a measurement provides additional confidence in a result’s validity. Its value accounts for all the factors which could possibly affect the result, such as adequacy of analyte definition, sampling, matrix effects and interferences, climatic conditions, variances in weights, volumes, and standards, analytical procedure, and random variation. Some national accreditation organizations require the use of an “expanded uncertainty”: which is the range within which the value of the measurand is believed to lie within at least a 95% confidence level with the coverage factor  $k=2$ .

**19.12.2** Uncertainty is not error. Error is a single value, the difference between the true result and the measured result. On environmental samples, the true result is never known. The measurement is the sum of the unknown true value and the unknown error. Unknown error is a combination of systematic error, or bias, and random error. Bias varies predictably, constantly, and independently from the number of measurements. Random error is unpredictable, assumed to be Gaussian in distribution, and reducible by increasing the number of measurements.

**19.12.3** The minimum uncertainty associated with results generated by the laboratory can be determined by using the Laboratory Control Sample (LCS) accuracy range for a given analyte. The LCS limits are used to assess the performance of the measurement system since they take into consideration all of the laboratory variables associated with a given test over time (except for variability associated with the sampling and the variability due to matrix effects). The percent recovery of the LCS is compared either to the method-required LCS accuracy limits or to the statistical, historical, in-house LCS accuracy limits.

**19.12.4** To calculate the uncertainty for the specific result reported, multiply the result by the decimal of the lower end of the LCS range percent value for the lower end of the uncertainty range, and multiply the result by the decimal of the upper end of the LCS range percent value for the upper end of the uncertainty range. These calculated values represent a 99%-certain range for the reported result. As an example, suppose that the result reported is 1.0 mg/l, and the LCS percent recovery range is 50 to 150%. The uncertainty range would be 0.5 to 1.5 mg/l, which could also be written as  $1.0 \pm 0.5$  mg/l.

**19.12.5** Alternatively, the information provided by the AHIA can be used to calculate uncertainty. This information can be found in the AIHA Laboratory Accreditation Program, LLC Policy Modules – Appendix G.

**19.12.6** In the case where a well recognized test method specifies limits to the values of major sources of uncertainty of measurement (e.g., 524.2, 525, etc.) and specifies the form of presentation of calculated results, no further discussion of uncertainty is required.

**19.12.7** The laboratory provides measurement uncertainty data only at client request. An additional fee is charged for the reporting of measurement uncertainty

### **19.13 Sample Reanalysis Guidelines**

Because there is a certain level of uncertainty with any analytical measurement, a sample re-preparation (where appropriate) and subsequent analysis (hereafter referred to as 'reanalysis') may result in either a higher or lower value from an initial sample analysis. There are also variables that may be present (e.g., sample homogeneity, analyte precipitation over time, etc.) that may affect the results of a reanalysis. Based on the above comments, the laboratory will reanalyze samples at a client's request with the following caveats. **Client specific Contractual Terms & Conditions for reanalysis protocols may supersede the following items.**

- Homogenous samples: If a reanalysis agrees with the original result to within the RPD limits for MS/MSD or Duplicate analyses, or within  $\pm 1$  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 Department Manager or Laboratory Director if unsure.

### **19.14 Control of Data**

The laboratory has policies and procedures in place to ensure the authenticity, integrity, and accuracy of the analytical data generated by the laboratory.

#### **19.14.1 Computer and Electronic Data Related Requirements**

The three basic objectives of our computer security procedures and policies are shown below. More detail is outlined in SOP PE-ADM-001 Computer Security. The laboratory is currently running the Element LIMS which is a 3<sup>rd</sup> party 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 Sequel Server / Access database which is an industry standard relational database platform. It is referred to as Database for the remainder of this section.

**19.14.1.1 Maintain the Database Integrity:** Assurance that data is reliable and accurate through data verification (review) procedures, password-protecting access, anti-virus protection, data change requirements, as well as an internal LIMS permissions procedure.

- LIMS Database Integrity is achieved through data input validation, internal user controls, and data change requirements.

- Spreadsheets and other software developed in-house must be verified with documentation through hand calculations prior to use. Cells containing calculations must be lock-protected and controlled.
- Instrument hardware and software adjustments are safeguarded through maintenance logs, audit trails and controlled access.

**19.14.1.2 Ensure Information Availability:** Protection against loss of information or service is ensured through scheduled back-ups, stable file server network architecture, secure storage of media, line filter, Uninterruptible Power Supply (UPS), and maintaining older versions of software as revisions are implemented.

**19.14.1.3 Maintain Confidentiality:** Ensure data confidentiality through physical access controls such as password protection or website access approval when electronically transmitting data.

#### **19.14.2 Data Reduction**

The complexity of the data reduction depends on the analytical method and the number of discrete operations involved (e.g., extractions, dilutions, instrument readings and concentrations). The analyst calculates the final results from the raw data or uses appropriate computer programs to assist in the calculation of final reportable values.

For manual data entry, e.g., Wet Chemistry, the data is reduced by the analyst and then verified by the Department Manager or alternate analyst prior to updating the data in LIMS. The spreadsheets, or any other type of applicable documents, are signed by both the analyst and alternate reviewer to confirm the accuracy of the manual entry(s).

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

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 and the laboratory SOP PE-QAD-009 Manual Integration / Data Integrity.

Analytical results are reduced to appropriate concentration units specified by the analytical method, taking into account factors such as dilution, sample weight or volume, etc. Blank correction will be applied only when required by the method or per manufacturer's indication; otherwise, it should not be performed. Calculations are independently verified by appropriate laboratory staff. Calculations and data reduction steps for various methods are summarized in the respective analytical SOPs or program requirements.

**19.14.2.1** All raw data must be retained in the worklist folder, computer file (if appropriate), and/or runlog. All criteria pertinent to the method must be recorded. The documentation is recorded at the time observations or calculations are made and must be signed or initialed/dated (month/day/year). It must be easily identifiable who performed which tasks if multiple people were involved.

**19.14.2.2** In general, concentration results are reported in milligrams per liter (mg/l) or micrograms per liter ( $\mu\text{g/l}$ ) for liquids and milligrams per kilogram (mg/kg) or micrograms per kilogram ( $\mu\text{g/kg}$ ) for solids. For values greater than 10,000 mg/l, results can be reported in percent, i.e., 10,000 mg/l = 1%. Units are defined in each lab SOP.

**19.14.2.3** In reporting, the analyst or the instrument output records the raw data result using values of known certainty plus one uncertain digit. If final calculations are performed external to LIMS, the results should be entered in LIMS with at least three significant figures. In general, results are reported to 2 significant figures on the final report.

**19.14.2.4** For those methods that do not have an instrument printout or an instrumental output compatible with the LIMS System, the raw results and dilution factors are entered directly into LIMS by the analyst, and the software calculates the final result for the analytical report. LIMS has a defined significant figure criterion for each analyte.

**19.14.2.5** The laboratory strives to import data directly from instruments or calculation spreadsheets to ensure that the reported data are free from transcription and calculation errors. For those analyses with an instrumental output compatible with the LIMS, the raw results and dilution factors are transferred into LIMS electronically after reviewing the quantitation report, and removing unrequested or poor spectrally-matched compounds. The analyst prints a copy of what has been entered to check for errors. This printout and the instrument's printout of calibrations, concentrations, retention times, chromatograms, and mass spectra, if applicable, are retained with the data file. The data file is stored in a monthly folder on the instrument computer; periodically, this file is transferred to the server and, eventually, to a tape file.

### **19.14.3 Logbook / Worksheet Use Guidelines**

Logbooks and worksheets are filled out 'real time' and have enough information on them to trace the events of the applicable analysis/task. (e.g. calibrations, standards, analyst, sample ID, date, time on short holding time tests, temperatures when applicable, calculations are traceable, etc.)

- Corrections are made following the procedures outlined in Section 12.
- Logbooks are controlled by the QA department. A record is maintained of all logbooks in the lab.
- Unused portions of pages must be Z'd out, signed and dated.
- Worksheets are created with the approval of the Department Manager / QA Manager at the facility. The QA Manager controls all worksheets following the procedures in Section 6.

### **19.14.4 Review / Verification Procedures**

Review procedures are outlined in the following SOPs to ensure that reported data are free from calculation and transcription errors, that QC parameters have been reviewed and evaluated before data is reported:

- PE-QAD-024 General Data Review
- PE-QAD-018 Use of Data Qualifiers
- PE-SMP-001 Sample Control
- PE-PMD-001 Data Reporting, Validation and Distribution

- PE-QAD-006 Logbook Documentation
- PE-QAD-007 Corrective Actions
- PE-QAD-022 Good Calibration Practices

The laboratory also has an SOP discussing Manual Integrations to ensure the authenticity of the data (SOP PE-QAD-009 Manual Integration / Data Integrity). The general review concepts are discussed below, more specific information can be found in the SOPs.

**19.14.4.1** The data review process at the laboratory starts at the Sample Control level. Sample Control personnel review chain-of-custody forms and input the sample information and required analyses into the laboratory LIMS program. The Project Managers perform final review of the chain-of-custody forms and inputted information.

**19.14.4.2** The next level of data review occurs with the Analysts. As results are generated, analysts review their work to ensure that the results generated meet QC requirements and relevant 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, initial and continuing calibration results, laboratory control samples, sample data, qualifiers and spike information are evaluated. Where calibration is not required on a daily basis, secondary review of the initial calibration results may be conducted at the time of calibration. Approximately 15% of all sample data from manual methods and from automated methods, all GC/MS spectra and all manual integrations are reviewed. For some methods, manual integrations are also electronically reviewed utilizing auditing software to help ensure compliance to ethics and manual integration policies. Issues that deem further review include the following:

- QC data are outside the specified control limits for accuracy and precision
- Reviewed sample data does not match with reported results
- Unusual detection limit changes are observed
- Samples having unusually high results
- Samples exceeding a known regulatory limit
- Raw data indicating some type of contamination or poor technique
- Inconsistent peak integration
- Transcription errors
- Results outside of calibration range

**19.14.4.3** Unacceptable analytical results may require reanalysis of the samples. Any problems are brought to the attention of the Laboratory Director, Project Manager, Quality Assurance Manager, or Department Manager for further investigation. Corrective action is initiated whenever necessary.

**19.14.4.4** The results are then entered or directly transferred into the computer database and a hard copy (or .pdf) is printed for the client.

**19.14.4.5** As a final review prior to the release of the report, the Project Manager reviews the results for appropriateness and completeness. This review and approval ensures that client requirements have been met and that the final report has been properly completed. The process includes, but is not limited to, verifying that chemical relationships are evaluated, COC is followed, cover letters/ narratives are present, flags are appropriate, and project specific requirements are met.

**19.14.4.6** Any project that requires a data package is subject to a tertiary data review for transcription errors and acceptable quality control requirements. The Project Manager then signs the final report. When complete, the report is sent out to the client.

**19.14.4.7** A visual summary of the flow of samples and information through the laboratory, as well as data review and validation, is presented in Figure 19-2.

#### **19.14.5 Manual Integrations**

Computerized data systems provide the analyst with the ability to re-integrate raw instrument data in order to optimize the interpretation of the data. Though manual integration of data is an invaluable tool for resolving variations in instrument performance and some sample matrix problems, when used improperly, this technique would make unacceptable data appear to meet quality control acceptance limits. Improper re-integrations lead to legally indefensible data, a poor reputation, or possible laboratory decertification. Because guidelines for re-integration of data are not provided in the methods and most methods were written prior to widespread implementation of computerized data systems, the laboratory trains all analytical staff on proper manual integration techniques using TestAmerica's Corporate SOP CA-Q-S-002 as the guideline for the laboratory's internal SOP PE-QAD-009 Manual Integration / Data Integrity.

- 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.
- Analysts shall not increase or decrease peak areas for the sole purpose of achieving acceptable QC recoveries that would have otherwise been unacceptable. The intentional recording or reporting of incorrect information (or the intentional omission of correct information) is against company principals and policy and is grounds for immediate termination.
- 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.
- All manual integrations receive a second level review. Manual integrations must be indicated on an expanded scale "after" chromatograms such that the integration performed can be easily evaluated during data review. Expanded scale "before" chromatograms are also required for all manual integrations on QC parameters (calibrations, calibration verifications, laboratory control samples, internal standards, surrogates, etc.) unless the laboratory has another documented corporate approved

procedure in place that can demonstrate an active process for detection and deterrence of improper integration practices.

**Figure 19-1. Example - Demonstration of Capability Documentation**

**TestAmerica Laboratories, Inc.  
Demonstration of Capability  
Authorization/Certification Statement**

Date:

Laboratory Name: TestAmerica Laboratories, Inc.  
Laboratory Address: 4625 East Cotton Center Boulevard, Suite 189  
Phoenix, AZ 85040

Analyst Name: Matrix: Media Type:

Method Number: SOP / Rev No.:

Analyte, or Class of Analytes or Measured Parameters:

We, the undersigned, CERTIFY that:

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 and/or the American Industrial Hygiene Association (AIHA) Accreditation Program, has met the Demonstration of Capability and is authorized to perform the above named analysis on the date listed.
2. The test method(s) was performed by the analyst identified on this certification.
3. A copy of the test method(s) and the SOP(s) are available for all personnel on-site.
4. The data associated with the demonstration of capability are true, accurate, and complete.
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.

Initial Demonstration Study (4 consecutive)  Ongoing DOC (AIHA every 6 months/NELAC annually)

Demonstrated by (Select one):

- BS / BSD (4 consecutive spikes)  RLV  
 BS / BSD (2 consecutive batches)  Desorption Efficiency Study  
 4 Consecutive QCS  MDL / MDLV (Circle one)  
 Side by Side Analysis/Supervisor Approval – when no spike is available  
 Acceptable Proficiency Testing Sample  PT/Work Order ID

\_\_\_\_\_  
Analyst Supervisor Name and Title Signature Date

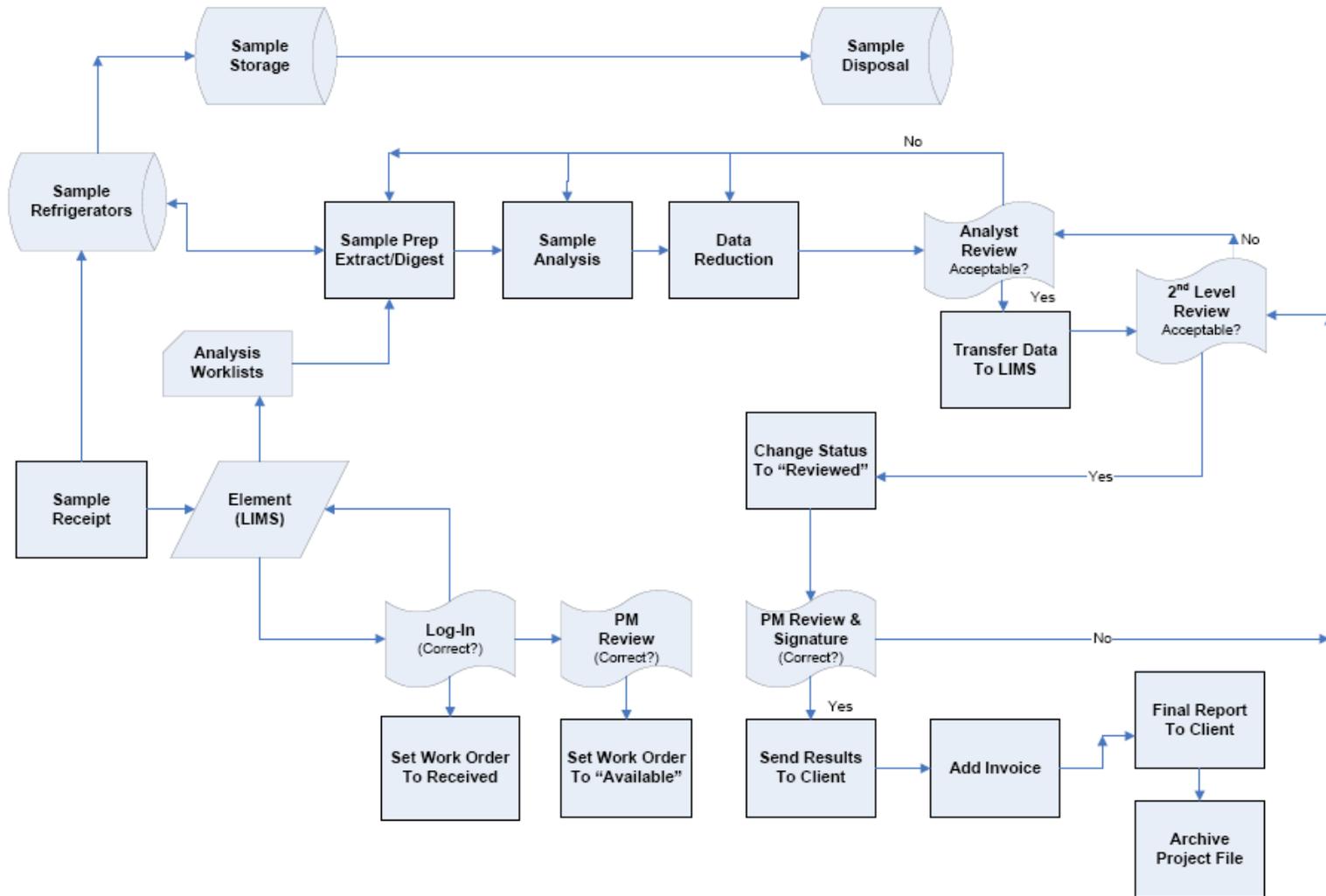
\_\_\_\_\_  
Quality Assurance Officer's Name Signature Date

This certification form must be completed each time a NELAC and/or AIHA demonstration of capability study is completed.

True: Consistent with supporting data.  
Accurate: Based on good laboratory practices consistent with sound scientific principles/practices.  
Complete: Includes the results of all performance testing.

Comments:

Figure 19-2. Example: Work Flow



## **SECTION 20. EQUIPMENT and CALIBRATIONS**

### **20.1 Overview**

The laboratory purchases the most technically advanced analytical instrumentation for sample analyses. Instrumentation is purchased on the basis of accuracy, dependability, efficiency and sensitivity. Each laboratory is furnished with all items of sampling, preparation, analytical testing and measurement equipment necessary to correctly perform the tests for which the laboratory has capabilities. Each piece of equipment is capable of achieving the required accuracy and complies with specifications relevant to the method being performed. Before being placed into use, the equipment (including sampling equipment) is calibrated and checked to establish that it meets its intended specification. The calibration routines for analytical instruments establish the range of quantitation. Calibration procedures are specified in the Corporate SOP CA-Q-S-005 Calibration Curves (General), laboratory SOPs and in SOP PE-QAD-022 Good Calibration Procedures. A list of laboratory instrumentation is presented in Table 20-1.

Equipment is only operated by authorized and trained personnel. Manufacturers' instructions for equipment use are readily accessible to all appropriate laboratory personnel.

### **20.2 Preventive Maintenance**

The laboratory follows a well-defined maintenance program to ensure proper equipment operation and to prevent the failure of laboratory equipment or instrumentation during use. This program of preventive maintenance helps to avoid delays due to instrument failure.

Routine preventive maintenance procedures and frequency, such as cleaning and replacements, should be performed according to the procedures outlined in the manufacturer's manual. Qualified personnel must also perform maintenance when there is evidence of degradation of peak resolution, a shift in the calibration curve, loss of sensitivity, or failure to continually meet one of the quality control criteria.

Table 20-2 lists examples of scheduled routine maintenance. It is the responsibility of each Technical/Department Manager to ensure that instrument maintenance logs are kept for all equipment in his/her department. Preventative maintenance procedures may be / are also outlined in analytical SOPs or instrument manuals. (Note: for some equipment, the log used to monitor performance is also the maintenance log. Multiple pieces of equipment may share the same log as long as it is clear as to which instrument is associated with an entry.)

Instrument maintenance logs are controlled and are used to document instrument problems, instrument repair and maintenance activities. Maintenance logs shall be kept for all major pieces of equipment. Instrument maintenance logs may also be used to specify instrument parameters.

- 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.
- Each entry in the instrument log includes the Analyst's initials, the date, a detailed description of the problem (or maintenance needed/scheduled), a detailed explanation of the solution or

maintenance performed, and a verification that the equipment is functioning properly (state what was used to determine a return to control. e.g. CCV run on 'date' was acceptable, or instrument recalibrated on 'date' with acceptable verification, etc.) must also be documented in the instrument records.

- When maintenance or repair is performed by an outside agency, service receipts detailing the service performed can be affixed into the logbooks adjacent to pages describing the maintenance performed. This stapled in page must be signed across the page entered and the logbook so that it is clear that a page is missing if only half a signature is found in the logbook.

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.

In the event of equipment malfunction that cannot be resolved, service shall be obtained from the instrument vendor manufacturer, or qualified service technician, if such a service can be tendered. If on-site service is unavailable, arrangements shall be made to have the instrument shipped back to the manufacturer for repair. Back up instruments, which have been approved, for the analysis shall perform the analysis normally carried out by the malfunctioning instrument. If the back up is not available and the analysis cannot be carried out within the needed timeframe, the samples shall be subcontracted.

If an instrument is sent out for service or transferred to another facility, it must be recalibrated and verified (including new initial MDL study) prior to return to lab operations.

### **20.3 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, thermal/pressure sample preparation devices and volumetric dispensing devices if quantitative results are dependent on their accuracy, as in standard preparation and dispensing or dilution into a specified volume. All raw data records associated with the support equipment are retained to document instrument performance.

#### **20.3.1 Weights and Balances**

The accuracy of the balances used in the laboratory is checked every working day, before use. All balances are placed on stable counter tops.

Each balance is checked prior to initial serviceable use with at least two certified ASTM type 1 weights spanning its range of use (weights that have been calibrated to ASTM type 1 weights may also be used for daily verification). ASTM type 1 weights used only for calibration of other weights (and no other purpose) are inspected for corrosion, damage or nicks at least annually and if no damage is observed, they are calibrated at least every 5 years by an outside calibration laboratory. Any weights (including ASTM Type 1) used for daily balance checks or

other purposes are recalibrated/recertified annually to NIST standards (this may be done internally if laboratory maintains "calibration only" ASTM type 1 weights).

All balances are serviced annually by a qualified service representative, who supplies the laboratory with a certificate that identifies traceability of the calibration to the NIST standards.

All balances are serviced annually by a qualified service representative accredited to ISO 17025, who supplies the laboratory with a certificate that identifies traceability of the calibration to NIST standards.

All of this information is recorded in logs, and the recalibration/recertification certificates are kept on file in QA. For additional information, reference laboratory SOP PE-QAD-016 Balance Calibration and Documentation.

### **20.3.2 pH, Conductivity, and Turbidity Meters**

The pH meters used in the laboratory are accurate to  $\pm 0.1$  pH units, and have a scale readability of at least 0.05 pH units. The meters automatically compensate for the temperature, and are calibrated with at least two working range buffer solutions before each use.

Conductivity meters are also calibrated before each use with a known standard to demonstrate the meters do not exceed an error of 1% or one umhos/cm.

Turbidity meters are also calibrated before each use. All of this information is documented in logs.

Consult pH and Conductivity, and Turbidity SOPs for further information.

### **20.3.3 Thermometers**

All thermometers are verified on at least an annual basis with a NIST-traceable thermometer. IR thermometers are verified semi- annually, digital thermometers are verified quarterly.

The mercury and digital NIST thermometers are recalibrated every five years (mercury) and one year (digital) (unless thermometer has been exposed to temperature extremes or apparent separation of internal liquid) by an approved outside service and the provided certificate of traceability is kept on file. The NIST thermometer(s) have increments of 0.1 degree (0.5 degree or less increments are required for drinking water microbiological laboratories), and have ranges applicable to method and certification requirements. The NIST traceable thermometers are 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 LIMS, instrument or method-specific logbooks or logsheets. More information on this subject can be found in the SOP PE-QAD-004 Thermometer Calibration.

#### **20.3.4 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. All of this information is documented in Daily Temperature Logsheets located in the QA office or in the Microbiology laboratory.

Ovens, waterbaths and incubators are monitored on days of use.

All of this equipment has a unique identification number, and is assigned a unique thermometer for monitoring.

Sample storage refrigerator temperatures are kept between 0°C and ≤6 °C.

Specific temperature settings/ranges for other refrigerators, freezers, ovens, waterbaths, and incubators can be found in method specific SOPs.

#### **20.3.5 Autopipettors, Dilutors, and Syringes**

Mechanical volumetric dispensing devices including burettes (except Class A Glassware and Glass microliter syringes) are given unique identification numbers and the delivery volumes are verified gravimetrically, at a minimum, on a quarterly basis.

For those dispensers that are not used for analytical measurements, a label is / can be applied to the device stating that it is not calibrated. Any device not regularly verified can not be used for any quantitative measurements.

Refer to SOP PE-QAD-002 Pipette Calibration for more information.

Micro-syringes are purchased from Hamilton Company. Each syringe is traceable to NIST. Hamilton attests to established accuracy and information is available on their website.

#### **20.3.6 Field Sampling Devices (Auto Samplers)**

Each Auto Sampler is assigned a unique identification number in order to keep track of the calibration. This number is also recorded on the sampling documentation.

The Auto Sampler is calibrated monthly by setting the sample volume to 100ml and recording the volume received. The results are filed in a logbook/binder. The Auto Sampler is programmed to run three (3) cycles and each of the three cycles is measured into a graduated cylinder to verify 100ml are received.

If the RSD (Relative Standard Deviation) between the 3 cycles is greater than 10%, the procedure is repeated and if the result is still greater than 10%, then the Auto Sampler is taken out of service until it is repaired and calibration verification criteria can be met. The results of this check are kept in a logbook/binder. (Please reference Table 20-5 for additional information.)

## **20.4      Instrument Calibrations**

Calibration of analytical instrumentation is essential to the production of quality data. Strict calibration procedures are followed for each method. These procedures are designed to determine and document the method detection limits, the working range of the analytical instrumentation and any fluctuations that may occur from day to day.

Sufficient raw data records are retained to allow an outside party to reconstruct all facets of the initial calibration. Records contain, but are not limited to, the following: calibration date, method, instrument, analyst(s) initials or signatures, analysis date, analytes, concentration, response, type of calibration (Avg RF, curve, or other calculations that may be used to reduce instrument responses to concentration).

Sample results must be quantitated from the initial calibration and may not be quantitated from any continuing instrument calibration verification unless otherwise required by regulation, method or program.

If the initial calibration results are outside of the acceptance criteria, corrective action is performed and any affected samples are reanalyzed if possible. If the reanalysis is not possible, any data associated with an unacceptable initial calibration will be reported with appropriate data qualifiers (refer to Section 12).

**Note:**      Instruments are calibrated initially and as needed after that and at least annually (the annual requirement does not apply to Isotope dilution).

### **20.4.1      Calibration Standards**

Calibration standards are prepared using the procedures indicated in the Reagents and Standards section of the determinative method SOP. If a reference method does not specify the number of calibration standards, a minimum of 3 calibration points (exception being ICP and ICP/MS methods) will be used.

Standards for instrument calibration are obtained from a variety of sources. All standards are traceable to national or international standards of measurement, or to national or international standard reference materials.

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).

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 at least the same number of significant figures used to report the data) must be reported as having less certainty, e.g., defined qualifiers or flags (additional information may be included in the case narrative). The exception to these rules is ICP methods or other methods where the referenced method does not specify two or more standards.

All initial calibrations are verified with a standard obtained from a second source and traceable to a national standard, when available (or vendor certified different lot if a second source is not available). This verification occurs immediately after the calibration curve has been analyzed, and before the analysis of any samples.

#### **20.4.1.1 Calibration Verification**

The calibration relationship established during the initial calibration must be verified initially and at least daily as specified in the laboratory method SOPs in accordance with the referenced analytical methods and in the 2009 TNI Standard and AIHA Industrial Hygiene Laboratory Accreditation Program (IHLAP). The process of calibration verification applies to both external standard and internal standard calibration techniques, as well as to linear and non-linear calibration models. Initial calibration verification is with a standard source secondary (second source standard), 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 at a different time or a different preparation would be considered a second source to the calibration standards, but continuing calibration verifications may use the same source standards as the calibration curve.

**Note:** The process of calibration verification referred to here is fundamentally different from the approach called "calibration" in some methods. As described in those methods, the calibration factors or response factors calculated during calibration are used to update the calibration factors or response factors used for sample quantitation. This approach, while employed in other EPA programs, amounts to a daily single-point calibration.

All target analytes and surrogates, including those reported as non-detects, must be included in periodic calibration verifications for purposes of retention time confirmation and to demonstrate that calibration verification criteria are being met, i.e., RPD, per 2009 TNI Std. EL-V1M4 Sec. 1.7.2.

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.

Generally, the initial calibrations must be verified at the beginning of each 12-hour analytical shift during which samples are analyzed. (Some methods may specify more or less frequent verifications). The 12-hour analytical shift begins with the injection of the calibration verification standard (or the MS tuning standard in MS methods). The shift ends after the completion of the analysis of the last sample, QC, or standard that can be injected within 12 hours of the beginning of the shift.

A continuing instrument calibration verification (CCV) must be repeated at the beginning and, for methods that have quantitation by external calibration models, at the end of each analytical batch. Some methods have more frequent CCV requirements see specific SOPs. Most Inorganic methods require the CCV to be analyzed after ever 10 samples or injections, including matrix or batch QC samples.

If the results of a CCV are outside the established acceptance criteria and analysis of a second consecutive (and immediate) CCV fails to produce results within acceptance criteria, corrective

action shall be performed. Once corrective actions have been completed & documented, the laboratory shall demonstrate acceptable instrument / method performance by analyzing two consecutive CCVs, or a new initial instrument calibration shall be performed.

Sample analyses and reporting of data may not occur or continue until the analytical system is calibrated or calibration verified. However, data associated with an unacceptable calibration verification may be fully useable under the following special conditions:

a). when the acceptance criteria for the CCV are exceeded high (i.e., high bias) and the associated samples within the batch are non-detects, then those non-detects may be reported with a footnote or case narrative explaining the high bias. Otherwise the samples affected by the unacceptable CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted; or

b). when the acceptance criteria for the CCV 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 CCV shall be re-analyzed after a new calibration curve has been established, evaluated and accepted.

Samples reported by the 2 conditions identified above will be appropriately flagged.

#### **20.4.1.2 Verification of Linear and Non-Linear Calibrations**

Calibration verification for calibrations involves the calculation of the percent drift or the percent difference of the instrument response between the initial calibration and each subsequent analysis of the verification standard. (These calculations are available in the laboratory method SOPs. Verification standards are evaluated based on the % Difference from the average CF or RF of the initial calibration or based on % Drift or % Recovery if a linear or quadratic curve is used.

Regardless of whether a linear or non-linear calibration model is used, if initial verification criterion is not met, then no sample analyses may take place until the calibration has been verified or a new initial calibration is performed that meets the specifications listed in the method SOPs. If the calibration cannot be verified after the analysis of a single verification standard, then adjust the instrument operating conditions and/or perform instrument maintenance, and analyze another aliquot of the verification standard. If the calibration cannot be verified with the second standard, then a new initial calibration is performed.

#### **20.5 Tentatively Identified Compounds (TICs) – GC/MS Analysis**

For samples containing components not associated with the calibration standards, a library search may be made for the purpose of tentative identification. The necessity to perform this type of identification will be determined by the purpose of the analyses being conducted. Data system library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. TICs must be identified as such when reported to the client.

**Note:** If the TIC compound is not part of the client target analyte list but is calibrated by the laboratory and is both qualitatively and/or quantitatively identifiable, it should not be reported as

a TIC. If the compound is reported on the same form as true TICs, it should be qualified and/or narrated that the reported compound is qualitatively and quantitatively (if verification in control) reported compared to a known standard that is in control (where applicable).

For example, the RCRA permit or waste delisting requirements may require the reporting of non-target analytes. Only after visual comparison of sample spectra with the nearest library searches may the analyst assign a tentative identification.

## 20.6 GC/MS Tuning

Prior to any GCMS analytical sequence, including calibration, the instrument parameters for the tune and subsequent sample analyses within that sequence must be set.

Prior to tuning/auto-tuning the mass spec, the parameters may be adjusted within the specifications set by the manufacturer or the analytical method. These generally don't need any adjustment but it may be required based on the current instrument performance. If the tune verification does not pass it may be necessary to clean the source or perform additional maintenance. Any maintenance is documented in the maintenance log.

**Table 20-1. Instrumentation List**

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
<b>Volatiles:</b>					
GCMS 1 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Hewlett Packard Agilent Tekmar Varian	6890 5973 3000 Archon	US00007754 US70810388 95325005 13472	1997	-
GCMS 2 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Hewlett Packard Hewlett Packard OI OI	5890 5971 4560 Archon	3033A30276 2950A00789 224071 13559	2001	-
GCMS 4 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Hewlett Packard Hewlett Packard OI OI	5890 5971 4560 Archon	3240G18320 3234A04143 N124460502 13025	2001	-
GCMS 6 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Agilent Agilent Tekmar Varian	6850 5973 3000 Archon	US00002193 US10440932 96055001 13624	2001	-
GCMS 7 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Agilent Agilent Tekmar Tekmar	5890 5972 3100 2016	3336A60504 3524A03129 US01281001 95298002	--	-
GCMS 9 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Hewlett Packard Hewlett Packard OI Analytical Varian	5890 5972 4660 Archon	- 3307A00428 D611466185P 14623	--	-

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
GCMS 13 Gas Chromatograph Mass Spectrometer P&T Concentrator Autosampler	Hewlett Packard Hewlett Packard Tekmar EST	5890 5972 3000 Centurion	3133A37877 3549A03207 97223016 CENTS120100709	--	-
GCMS 10 Gas Chromatograph Mass Spectrometer Autosampler Preconcentrator Canister Cleaner Dynamic Diluter (Shared with IH)	Agilent Agilent Entech Entech Entech Entech	6890 5973 7032L 7100 3100 4600	US0039506 US03960554 0043 0162 0103 0041	--	-
GCMS 11 Gas Chromatograph Mass Spectrometer Autosampler Preconcentrator Canister Cleaner (Shared with IH)	Agilent Agilent Entech Entech Entech	6890N 5973 7032L 7100 3100	US10133093 US10461255 0061 0259 0155	--	-

**Semi-Volatiles:**

Gas Chromatograph 1: (shared with IH) ALS Tower ALS Tray Controller Box FPD1 FPD2	Agilent Agilent Agilent Agilent Agilent Agilent	5890 18593B 18596B 18594B 19256A 19256A	2750A18397 3108A25342 3106A24228 3018A22248 NA NA	1990	--
Gas Chromatograph 2 ALS Tower ALS Tray Controller Box ECD1 ECD2	Agilent Agilent Agilent Agilent Agilent	5890 Series II 18593B 18596C G1512A G1223A G1223A	3108A34049 3508A41897 US30608322 CN00003596 K0668 F5885	1988	--
Gas Chromatograph 3 Linkbox ALS Concentrator PID Lam Power Source ECD/PID	Agilent OI Analytical EST Analytical EST Analytical OI Analytical OI Analytical	5890 Series II 600 Series CentWS Encon EV 4430 NA	3336A51039 5192110120 CENTS138022210 EV239012910 B348430309 NA	1995 2010 1995	
Gas Chromatograph 4 Concentrator ALS PID Lamp Power Source ECD/PID	Agilent OI Analytical OI Analytical OI Analytical OI Analytical	5890 Series II 4560 MPM16 4430 NA	2950A26451 D309335 91-369 91-171 NA	1995	
Gas Chromatograph 5 ALS Tower ALS Tray Controller Box FID MACH	Agilent Agilent Agilent Agilent Agilent Agilent (former RVM Scientific	5890 18593B 18596M 18594B NA NA	2643A9891 3042A23537 3251A30857 3239A30053 NA NA	1990 2005	
MACH Power Box	Agilent	LTMA58/A68PS	G E-01		--

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
Gas Chromatograph 7 (shared with IH) ALS Tower ALS Tray u-ECD1 u-ECD2	Agilent Agilent Agilent Agilent Agilent	6890 G2613 G2614A G2397A G2397A	US00000197 CN33832614 US91605057 U1290 U6313	2003	--
Gas Chromatograph 11 ALS Tower ALS Tray Controller Box ECD2	Agilent Agilent Agilent Agilent Agilent	5890A 18593B 18596B 18594A G1223A	3140A38412 3048A24494 3246A30486 2929A15556 F6883	1990	--
Gas Chromatograph 12 ALS Tower ALS Tray Controller Box u-ECD1 u-ECD2	Agilent Agilent Agilent Agilent Agilent Agilent	6890 G1513A 18596M G1512A G2397A G2397A	US00001438 US05012072 US30608322 3530A02441 U2666 U0495	1997	
High-Performance Liquid Chromatograph 3 ALS COL COM FLD Dual-DAD DEGASSER QUAD PUMP	Agilent Agilent Agilent Agilent Agilent Agilent	G131A G1316A G1321A G1315B G1379A G1311A	DE23922078 DE23930798 DE92001260 DE30518838 JP13205067 DE23920683	2004  2009 2004	New
GCMS12 Gas Chromatograph Mass Spectrometer ALS Tower ALS Tray	Agilent Agilent Agilent Agilent	G1530A G2578A G2613A G2614A	US00040094 US21853018 US93909504 US92905661	2000	--
GCMS14 Gas Chromatograph Mass Spectrometer ALS Tower ALS Tracy	Agilent Agilent Agilent Agilent	G1530N G3172A G2913A G2614A	CN10430038 US54431689 CN81247973 CN80647378	2004	--

**Extractions:**

Accelerated Solvent Extractor #3	Dionex	200	99030374	2003	--
Accelerated Solvent Extractor #4	Dionex	ASE 200	97040459	2002	--
Accelerated Solvent Extractor #5	Dionex	ASE 200	3040683	2005	--
Accelerated Solvent Extractor #6	Dionex	ASE 200	03110492	2011	New
Accelerated Solvent Extractor #7	Dionex	ASE 200	03110490	2011	New
Nanopure Water System	Barnstead	4741	747940357923	1998	--
Muffle Furnace	Thermolyne	62700	627970243372	1999	--
BL006: Analytical Balance (Shared with IH)	Sartorius	CP225D	14204830	--	--
Refrigerator Recirculator	Neslab	CFT-75	87KML60200-20	1999	--

**Metals:**

ICP03 (Shared with IH)	Perkin Elmer	5300DV	077C7070202	2006	New
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Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
ICP02 (Shared with IH)	Perkin Elmer	5300DV	077N6041401	2006	New
ICP/MS01 (Shared with IH)	Perkin Elmer	ELAN 6100	G2700107	2001	New
ICP/MS02 (Shared with IH)	Thermo	X - Series	X0376		Used
Mercury Analyzer 02 (Shared with IH)	Perkin Elmer	FIMS 100	101S4080502	2004	New
Mercury Analyzer 03	Perkin Elmer	FIMS 400	135951	2000	--
Hot Block Digestor A (Shared with IH)	Environ. Expr.	SC154	1423CEC1098	--	--
Hot Block Digestor C (Shared with IH)	Environ. Expr.	SC154	526CEC0747		
Hot Block Digestor D (Shared with IH)	Environ. Expr.	SC154	2484CEC1296		
Hot Block Digestor E (Shared with IH)	Environ. Expr.	SC154	1423CEC1099		
Hot Block Digestor F (Shared with IH)	Environ. Expr.	SC154	424CEC0592	2003	New
Hot Block Digestor B (Shared with IH)	Environ. Expr.	SC154	1944CEC1006	2001	New
TCLP Rotator	Environ. Expr.	--	--	--	--
TCLP Rotator	Environ. Expr.	--	--	--	--

<b>Wet Chemistry:</b>					
Ion Chromatograph 3	Dionex	ICS1000	04050018	2004	New
Autosampler	Dionex	AS40	95090256	1995	--
Ion Chromatograph 4	Dionex	ICS2000	05090476	2005	New
Autosampler	Dionex	AS40	05090256	2005	New
Ion Chromatograph 5	Dionex	ICS2000	04050699	2006	New
Autosampler	Dionex	AS40	94110334	1994	--
Ion Chromatograph 6	Dionex	ICS2000	07020086	2007	New
Autosampler	Dionex	AS40	04040861	2004	New
PC Titrator 1	ManTech	PC-1000-102/4	MS-0A4-357	2003	New
PC Titrate Interface Module	ManTech	PC-1300-475	MS-0D4-634	2003	New
Titra-Sip Titration Module	ManTech	PC-1104-00	MS-9B9-399	2003	New
Burivar 1/2 Buret Module	ManTech	PC-1000-408	MS-0J3-167	2003	New
Titra-Rinse/A Module	ManTech	PC-1000-408	MS-0J3-167	2003	New
Conductivity Meter	Jenway	4510	1106	2003	New
Autosampler	ManTech	--	270J3XB590	2003	New
PC Titrator 2	ManTech	PC-1075-00	MS-1H0-105	2010	New
Titra-Sip SA Interface Module	ManTech	PC-1075-00	MS-1H0-105	2010	New
Titra-Sip Titration Module	ManTech	PC-1300-475	MS-1F0-817	2010	New
Titra-Rinse/A Module	ManTech	PCM-1000-470	MS-0F4-191	2010	New
Titra-Rinse/A Module	ManTech	PCM-1000-400	MS-0J2-535	2010	New
Autosampler	ManTech	PC-1000-681	190A3032	2010	New

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
BOD Auto-Analyzer Interface Module	ManTech	PC-1085-00	MS-1B0-136	2010	New
Titra-Rinse/A Module	ManTech	PC-1000-480	MS-0F5-243	2010	New
Titra-Rinse/A Module	ManTech	PC-1000-443	MS-1A0-111	2010	New
Sensor & Stirrer Control	ManTech	PB-10030	MS-1B0-106	2010	New
Inhibitor Pump	ManTech	PC-1000-475	MS-1B0-118	2010	New
Rinse Pump	ManTech	PC-1000-470	MS-1B0-113	2010	New
Dissolved Oxygen Meter 2	YSI	52CE	03J0616	2003	New
Autosampler	ManTech	PBM-1000-688	260A8N025	2010	New
Total Organic Carbon Analyzer	Shimadzu	TOC-V-CSH	40D91227	2002	--
Autosampler	Shimadzu	ASI-V	H52104100104	2002	--
Pensky-Martins Flash Tester	Fisher Scientific	--	20800023	--	--
UV-Vis Spectrophotometer	Shimadzu	UV Pharma Spec 1700	A11024136179 CS	2003	New
Turbidimeter	HF Scientific	Micro-100	200702190	2007	New
Dissolved Oxygen Meter	YSI	5000	99D0533	--	--
pH / ISE Meter 3	Thermo/Orion	710A	060237	--	--
pH / ISE Meter 4	Orion	420A	014395	--	--
pH / ISE Meter 5	Orion	420A	24440	--	--
Conductivity / pH Meter	Hach	HQ30d	060600000983	2006	New
Conductivity Meter	Control Company		98291048	--	--
COD Block Reactor 3	Hach	45600-00	010700022054	--	--
COD Block Reactor 4	Hach	LTG082.54.42001	1218899	2006	NEW
COD Block Reactor	Hach	LTG082.54.44001	1156212	2007	NEW
TKN Digestion System Block Controller	Aim Lab Aim Lab	AIM600 AIM 600	4904A14055 4906A14087	2008 2008	New New
Cyanide MIDI - Distillation System	Lab Crest	110-10-REG	SNA4U0072	--	--
Cyanide MIDI - Distillation System	Lab Crest	110-10-R	A9P0209	--	--
Hot Block Digestor	Environ. Expr.	SC100	615CEC0860	--	--
BOD Incubator 04	VWR	2020	05103004	--	--
BOD Incubator 05	Revco	--	--	--	--
BOD Incubator 06	Lab Line	3554-40	1097-001	--	--
Analytical Balance 009	Ohaus	Scout Pro SP601	7122181160	--	--
Analytical Balance 012	Mettler Toledo	AX205	1122481540	--	--
Analytical Balance 017	Sartorius	A1205-**D20	39050003	--	--
Analytical Balance 019	Mettler	AE260-5	G31175	--	--
Drying Oven 004	VWR	1305U	0705590	--	--
Drying Oven 006	VWR	1320	0800599	--	--
Drying Oven 007	Blue M	OV-500C-2	OV3-24912		
Drying Oven 010	Fisher Scientific	630G	20400063	--	--
Muffle Furnace 01	ThermoLyne	62700	--	--	--
E-Pure System	Barnsted	D4641	1090050246637	--	--
Centrifuge	Beckman	TJ-6	0A058	--	--
Reciprocal Shaker	Lab Line	3506	0793-0453	--	--
Mini Vortexer	VWR	VM3000	25347	--	--

**Microbiology :**

Quanti Tray Sealer	Idexx	2020	--	2002	Used
Quanti Tray Sealer	Idexx	QT001	4120	2005	Used
Water Bath 8	Precision	51221033	601121635	2002	New

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
Water Bath 6	Boekel	GD100L	GL054300	--	New
Water Bath 5	Boekel	GD100L	GL0450003	--	New
Incubator 4	Thermo	3973	304764	2005	New
Incubator 5	VWR	1915	--	1992	New
Mini Vortexer	VWR	VM3000	060223015	2003	--
Analytical Balance	Ohaus	Adventuer-Pro	8026421198	2005	New
Microscope	Nikon	Nme	135387	--	New
Microscope	Leica	Zoom 2000	132DEZ	--	New

<b>Industrial Hygiene:</b>					
HPLC2 High Performance Liquid Chromatograph 1100 ALS	Agilent	G1313A G1322A G1311A G1316A G1321A G1315B	DE91610196 JP73020320 DE11114347 DE91612722 DE92001665 DE11112225	--	--
HPLC4 High Performance Liquid Chromatograph 1100 ALS / LCQ Advantage Mass Spectrometer w/ DAD and Fluorescence Detectors	Agilent / Thermo -Finnigan	G1313A G1322A G1311A G1316A G1321A G1315A LCQADV	DE11115352 JP05029135 DE91608229 DE11120753 DE11103117 DE91607422 LAD00192	--	--
GC1: Controller 7673 Autosampler Injector 7673 Gas Chromatograph w/ dual FPD Detectors (Shared with SVOA)	Agilent	7673 18596 B 7673 5890	3018A22248 3106A24228 3108A25342 7750A18397	-- -- -- 1990	--
GC14: Controller Autosampler Injector Gas Chromatograph w/ dual FID Detectors	Agilent	7673 18596 B 7673 5890 Series II	3007A20952 3201A27340 3237A32148 3140A39271	-- -- -- --	--
GC13: Controller Injector Gas Chromatograph w/ FID and TCD Detectors	Agilent	7673 7673 5890 Series II	3113A25897 3048A24489 3140A38303	-- -- -- --	--
GC9: Controller Autosampler Injector Gas Chromatograph w/ FID Detector	Agilent	7673 18596 B 7673 5890 Series II	3251A30932 3334A32981 3120A26800 3118A35369	-- -- -- --	--
GC7: Autosampler Injector Gas Chromatograph w/ dual ECD Detectors (Shared with SVOA)	Agilent	G2614A 7683 6890	US91605057 CN33832614 US00000197	-- -- -- --	--
GC-MS 8: Gas Chromatograph Mass Spectrometer ATD	Hewlett Packard Agilent Perkin Elmer	6890 5973 TurboMatrix 650	3235A44760 3329A00483 TD650L0605128		2010 Used

Instrument Type	Manufacture	Model Number	Serial Number	Year Put into Service	Condition When Received
GCMS 10 Gas Chromatograph Mass Spectrometer Autosampler Preconcentrator Canister Cleaner Dynamic Diluter (Shared with VOA)	Agilent Agilent Entech Entech Entech Entech	6890 5973 7032L 7100 3100 4600	U50039506 U503960554 0043 0162 0103 0041		
GCMS 11 Gas Chromatograph Mass Spectrometer Autosampler Preconcentrator Canister Cleaner (Shared with VOA)	Agilent Agilent Entech Entech Entech Entech	6890 5973 7032L 7100 3100 4600	U50039506 U503960554 0043 0162 0103 0041		
ICP03 (Shared with Metals)	Perkin Elmer	5300DV	077C7070202	2006	New
ICP02 (Shared with Metals)	Perkin Elmer	5300DV	077N6041401	2006	New
ICP/MS01 (Shared with Metals)	Perkin Elmer	ELAN 6100	G2700107	2001	New
ICP/MS02 (Shared with Metals)	Thermo	X - Series	X0376		Used
Mercury Analyzer 02 (Shared with Metals)	Perkin Elmer	Fims 100	101S4080502	2004	New
Hot Block Digester A (Shared with Metals)	Environ. Expr.	SC154	1423CEC1098	--	--
Hot Block Digester B (Shared with Metals)	Environ. Expr.	SC154	1944CEC1006	--	--
Hot Block Digester C (Shared with Metals)	Environ. Expr.	SC154	526CEC0747		
Hot Block Digester D (Shared with Metals)	Environ. Expr.	SC154	2484CEC1296		
Hot Block Digester E (Shared with Metals)	Environ. Expr.	SC154	1423CEC1099		
Hot Block Digester F (Shared with Metals)	Environ. Expr.	SC154	424CEC0592	2003	New
IC2: Ion Chromatograph Interface AS-40 Autosampler	Dionex PE Nelson Dionex	DX-120 900 AS-40	97070800 1036512763 94120305	-- -- --	Used
IC7: Ion Chromatograph Pump TC VWD Pneumatic Controller Autosampler	Dionex	ICS 3000 ICS 3000 ICS 3000 ICS 3000 PC-10 AS-40	-- 08050969 08041101 08050957 063334 08051080	2008 2008 2008 2008 2008 2008	New New New New New New
SPEC2: Spectrophotometer	Turner	SP-830	1102980604474	--	--
BL006 : Analytical Balance (Shared with SVOA)	Sartorius	CP225D	14204830	--	--
BL020: Analytical Balance	Denver Instrument	XL-3100	0079735	2010	Used

**Table 20-2. Example: Schedule of Routine Maintenance**

Instrument	Procedure	Frequency
Mercury Analyzer	Check tubing for wear Fill rinse tank with 10% HCl Insert clean drying tube filled with Magnesium Perchlorate Fill reductant bottle with 10% Stannous Chloride	Daily Daily Daily Daily
ICP	Check pump tubing Check liquid argon supply Check fluid level in waste container Check filters Clean or replace filters Check torch Check sample spray chamber for debris Clean and align nebulizer Check entrance slit for debris Change printer ribbon Replace pump tubing	Daily Daily Daily Weekly As required Daily Monthly Monthly Monthly As required As required
ICP MS	Change pump tubing Clean torch Check / clean nebulizer Clean cones Check air filters Check multiplier voltages & do cross calibration Replace sample uptake tubing Check rotary pump oil Check oil mist filters Check chiller water level	Weekly Weekly Weekly Daily Weekly Weekly Monthly Monthly Monthly Monthly
UV-Vis Spectrophotometer	Clean ambient flow cell Precision check/alignment of flow cell Wavelength verification check	As required As required Semi-annually
Auto Analyzers	Clean sampler Check all tubing Clean inside of colorimeter Clean pump well and pump rollers Clean wash fluid receptacle Oil rollers/chains/side rails Clean optics and cells	Daily Daily Daily Quarterly Weekly Weekly Quarterly
Hewlett Packard GC/MS	Ion gauge tube degassing Pump oil-level check Pump oil changing Analyzer bake-out Analyzer cleaning Resolution adjustment COMPUTER SYSTEM AND PRINTER: Air filter cleaning Change data system air filter Printer head carriage lubrication Paper sprocket cleaning Drive belt lubrication	As required Monthly Annually As required As required As required As required As required As required As required As required As required

Instrument	Procedure	Frequency
Gas Chromatograph	Compare standard response to previous day or since last initial calibration Check carrier gas flow rate in column  Check temp. of detector, inlet, column oven Septum replacement Check system for gas leaks with SNOOP  Check for loose/frayed wires and insulation ½" Bake injector/column Change/remove sections of guard column Replace connectors/liners Change/replace column(s)	Daily  Daily via use of known compound retention Daily As required W/cylinder change as required Monthly As Required As Required As Required As Required
Electron Capture Detector (ECD)	Detector wipe test (Ni-63) Detector cleaning	Semi-annually As required
Flame Ionization Detector (FID)	Detector cleaning	As required
Flame Photoionization Detector (FPD)	Clean and/or Replace Lamp	As required
Photoionization Detector (PID)	Change O-rings Clean lamp window	As required As required
HPLC	Change guard columns Change lamps Change pump seals Replace tubing Change fuses in power supply Filter all samples Change autosampler rotor/stator	As required As required Semi-annually or as required As required As required Daily As required
Balances	Class "S" traceable weight check Clean pan and check if level Field service	Daily, when used Daily At least Annually
Conductivity Meter	0.01 M KCl calibration Conductivity cell cleaning	Daily As required
Turbidimeter	Check light bulb	Daily, when used
Deionized/Distilled Water	Conductivity Point Sources Daily conductivity check Check deionizer light Monitor for VOA's System cleaning Replace cartridge & large mixed bed resins	Water Quality Daily Daily As required As required
Drying Ovens	Temperature monitoring Temperature adjustments	Daily As required
Refrigerators/Freezers	Temperature monitoring Temperature adjustment Defrosting/cleaning	Daily As required As required
Vacuum Pumps/Air Compressor	Drained Belts checked Lubricated	As required As required Semi-annually

Instrument	Procedure	Frequency
pH/Specific Ion Meter	Calibration/check slope Clean electrode	Daily As required
BOD Incubator	Temperature monitoring Coil and incubator cleaning	Daily Monthly
Water baths	Temperature monitoring Water replaced	Daily Monthly or as needed

**Table 20-3. Preventative Maintenance for Laboratory Equipment**

Instrument/ Equipment Type	Preventative Maintenance	Frequency
<b>Gas Chromatograph</b>	Replace Gas line dryers and filters	As needed
	Replace Gas cylinders	As needed
	Check or adjust column gas flow and/or detector make-up flow	As needed
	Replace Injection port Septa	As needed
	Replace Injection port liners/re-silicone liners	GC, As needed; GC/MS, Daily
	Replace Injection port liner o-ring	GC, As needed; GC/MS, Daily
	Replace inlet seal and ring	GC, As needed, GC/MS, Daily
	Replace column ferrules	GC, As needed; *
	Clip column (injector and detector end)	GC, As needed; GC/MS, Daily
	Replace syringes on autosamplers	As needed
	Replace heated-zones heaters and sensors	As needed
	Replace inlet assembly	As needed
	Empty solvent rinse and solvent rinse-waste vials (on autosampler tower)	Daily or as needed
	Replace column	As needed
<b>Flame Ionization Detector (FID)</b>	Clean/replace jet	As needed
	Clean collector	As needed
	Check and/or adjust gas flows	As needed
	Replace graphite ferrule	After each cleaning (OI detectors only)
<b>Photoionization Detector (PID)</b>	Clean window	As needed
	Replace o-ring seat	As needed
	Replace Lamp	As needed
	Check and/or adjust gas flows	As needed
	Adjust Lamp power supply intensity	As needed
<b>Flame Photometric Detector (FPD)</b>	Clean mirrors/lenses	As needed
	Replace mirrors/lenses	As needed
	Replace o-rings	As needed
<b>Mass Spectrometer (MS)</b>	Clean source, replace source parts, replace filaments	As needed
	Clean analyzer	As needed

Instrument/ Equipment Type	Preventative Maintenance	Frequency
	Replace electron multiplier	As needed
	Clean or replace glass jet separator, replace transfer line from jet separator to MS	As needed
	Change rough pump oil	As needed
<b>Purge and Trap Equipment</b>	Refill rinse water supply/Empty rinse water waste	Weekly or as needed
	Refill spiking solutions vials	As needed
	Rinse sparge tubes	Daily
	Clean or replace 6-port valve	As needed
	Replace Transfer lines (from Autosampler to LSC and from LSC to GC)	As needed
	Adjust gas flows and pressures	As needed
	Perform leak check	As needed
<b>High Pressure Liquid Chromatography (HPLC)</b>	Calibrate Detector	As needed
	Replace pre-column filter	As needed
	Refill Solvent reservoirs	Daily or as needed
	Reverse column and rinse with solvents	Daily or as needed*
	Replace column	As needed
	Clean solvent reservoir filters	As needed
	Replace Guard Column	As needed
	Replace solvent reservoir frits	As needed
	Replace ball-valve cartridges on high pressure pump	As needed*
	Replace DAD flow cell windows	As needed*
	Check system solvent pressure	Daily
<b>Inductively Coupled Plasma, Atomic Emission Spectrometer (ICP-AES)</b>	Replace Peristaltic pump tubing	As needed
	Clean autosampler, change tubing	As needed
	Clean nebulizer and torch assembly	As needed
	Replace nitrogen and argon tanks	As needed
	Refill rinse water receptacle	Daily
	Empty waste receptacle	Daily
	Check for internal standard and sample flow through peristaltic pump tubing	As often as possible
	Replace internal standard solution receptacle	As needed
	Operate and check vents	Daily
	Perform Hg alignment	Daily
	Check water level and water filter on recirculating-cooling unit, refill and replace filter	Check daily, refill and replace as needed
	Check purge windows	Daily, replace as needed
	Replace nebulizer and o-rings	As needed
	Replace torch	As needed
Drain air compressor	Weekly	

<b>Instrument/ Equipment Type</b>	<b>Preventative Maintenance</b>	<b>Frequency</b>
	Replace mixing chambers	As needed
	Clean or replace air filters	Weekly
	Check pneumatic filters	Weekly, replace as needed
	Perform wave calibration (UV and Vis)	Quarterly*
<b>Mercury Analyzer</b>	Change Argon supply tank	As needed
	Change drying tube	Daily or as needed
	De-clog drying tube and/or reductant tubing	Daily or as needed
	Change system tubing	2-3 weeks
	Rinse tubing prior to operation and following operation	Daily
	Clean optical cell	As needed (when aperture is out of line)
<b>pH Meters</b>	Clean or replace electrode	As needed
	Refill electrode electrolyte	As needed
<b>Balance</b>	Clean pan and platform	After each use
	Check Level bubble	Daily
	Check calibration	Daily
	Check sensitivity	Weekly
	Cleaning and calibration by authorized service	Annually
<b>Conductivity Meter</b>	Clean probe	As needed
<b>Dissolved Oxygen Meter</b>	Replace membrane	As needed
	Clean probe	As needed
<b>ZHE vessels</b>	Replace o-rings and screens	As needed
<b>ZHE and TCLP Tumblers</b>	Check Rotation Rate	Monthly
<b>Spectrophotometers</b>	Clean and check tubing	As needed
<b>Burettes and Pipettes</b>	Clean and check calibration	Quarterly
<b>Thermometers</b>	Check calibration	Annually, Quarterly for Digitals and IR Thermometer
<b>Ovens</b>	Check and/or adjust temperature, record temperature on log sheet	Daily
<b>Refrigerators and Freezers</b>	Check and/or adjust temperature, record temperature on log sheet	Daily
	Defrost freezers	As needed

Table 20-4. Periodic Calibrations

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
<b>Analytical Balance</b>	Accuracy determined using NIST calibrated weights.  Minimum of 2 standards bracketing the weight of interest.  Annually inspected and calibrated by ISO accredited firm.	Daily	See logbook	Clean, check level, insure lack of drafts, and that unit is warmed up, recheck. If fails, call service or replace.
<b>Top Loading Balance</b>	Accuracy determined using NIST calibrated weights.  Minimum of 2 standards bracketing the weight of interest.  Annually inspected and calibrated by ISO accredited firm.	Daily	See logbook	Clean. If fails, call service or replace.
<b>NIST Weights</b>	Accuracy determined by accredited weights and measurement laboratory.	5 year	As per certificate.	Replace.
<b>Working Weights</b>	Examine for wear, compare against NIST weights.	Annually	ASTM Type 1, Class 1 or 2 standards	Replace.
<b>NIST-Traceable Thermometer</b>	Accuracy determined by accredited weights and measurement laboratory.	5 years	As per certificate.	Replace.
<b>Working Thermometers</b>	Against NIST-traceable thermometer	Yearly (or more frequently e.g. digital are checked quarterly) at appropriate temperature range for intended use	$\pm 1.0^{\circ}\text{C}$	Replace.

<b>Instrument</b>	<b>Type of Calibration/ Number of Standards</b>	<b>Frequency</b>	<b>Acceptance Limits</b>	<b>Corrective Action</b>
<b>InfraRed Temperature Guns</b>	Against calibrated liquid thermometer at ambient and storage temps	Daily	$\pm 0.5^{\circ}\text{C}$	Repair/replace.
	Against NIST-traceable thermometer	Semi-annually at appropriate temperature range for intended use.	$\pm 1.0^{\circ}\text{C}$	Repair/replace.
<b>Dial-type Thermo-meters</b>	Against NIST-traceable thermometer	Quarterly at appropriate temperature range for intended use.	$\pm 1.0^{\circ}\text{C}$	Replace.
<b>Refrigerator</b>	Temperature checked using NIST-traceable thermometer.	Daily. If out of range, recheck in two hours.	$0 \text{ to } \leq 6^{\circ}\text{C}$	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify Department Manager.
<b>Freezer</b>	Temperature checked using NIST-traceable thermometer	Daily. If out of range, recheck in two hours.	$\leq (-10)^{\circ}\text{C}$	Adjust. Repair. While waiting for repair, seal door, attach "Out of Service" sign, move items to functional unit. Notify Department Manager.
<b>Oven</b>	Temperature checked using NIST-traceable thermometer.	When in use.	$104 \pm 1^{\circ}\text{C}$ (drying) $180 \pm 2^{\circ}\text{C}$ (TDS); or as per method.	Adjust. Replace.
<b>Incubator</b>	Temperature checked using NIST-traceable thermometer.	When in use. For microbiology, twice daily when in use.	BOD: $20 \pm 1.0^{\circ}\text{C}$ Micro: $35 \pm 0.5^{\circ}\text{C}$	Adjust. Replace.
<b>Water Bath</b>	Temperature checked using NIST-traceable thermometer.	When in use.	See analytical SOP.	Adjust. Replace.

Instrument	Type of Calibration/ Number of Standards	Frequency	Acceptance Limits	Corrective Action
<b>Volumetric Dispensing Devices (Eppendorf® pipette, automatic dilutor or dispensing devices)</b>	One delivery by weight. Using DI water, dispense into tared vessel. Record weight with device ID number.  See SOP.	Monthly	± 2% Calculate accuracy by dividing weight by stated volume times 100 for percent.	Adjust. Replace.
<b>Glass Microliter Syringes</b>	None	Hamilton syringes are ordered with a certificate attesting to their accuracy.	Re-verified.	Replaced.
<b>Conductivity Meter</b>	Cell impedance calibrated with three KCl standards.	Each use.	$r \geq 0.99$	Recalibrate.
<b>Nanopure Water</b>	Check in-line conductivity meter on system with conductivity meter in Inorganics Department.	Each day of use.	$<10 \mu\text{mhos}/\text{cm}^2$	Record in logbook. Report discrepancies to the Department Manager.
	Check for compliance with Standard Methods reagent water requirements	Monthly	Ammonia $<.01\text{mg}/\text{L}$ Res. Cl $<0.01 \text{mg}/\text{L}$ pH 5.5 – 7.5 SU TOC $<1 \text{mg}/\text{L}$ HPC $<1000$ CFU/mL	Record in logbook. Report discrepancies to the Department Manager.

Table 20-5. Preventative Maintenance for Field Equipment

Instrument/ Equipment Type	Activity	Frequency	Maintenance
<b>Automatic Sampler – ISCO 3710/3910</b>	Check tubing and connections through pump head	Before and after use	Replace tubing when necessary
	Check battery power and program	Before and after use	Replace battery when necessary
	Clean tubing in pump head	After each use	Replace pump head tubing when necessary
	Clean tubing for sample collection	After each use	Not applicable
	Check functionality – manual sample; program sample	Prior to use	Not applicable
	Check sample container for breakage, etc.	Prior to use	Replace if needed

## **SECTION 21. MEASUREMENT TRACEABILITY**

### **21.1 Overview**

Traceability of measurements shall be assured using a system of documentation, calibration, and analysis of reference standards. Laboratory equipment that are peripheral to analysis and whose calibration is not necessarily documented in a test method analysis or by analysis of a reference standard shall be subject to ongoing certifications of accuracy. At a minimum, these must include procedures for checking specifications of ancillary equipment: balances, thermometers, temperature, Deionized (DI) and Reverse Osmosis (RO) water systems, automatic pipettes and other volumetric measuring devices (Refer to Section 20.3). With the exception of Class A Glassware and Glass microliter syringes, quarterly accuracy checks are performed for all mechanical volumetric devices. Wherever possible, subsidiary or peripheral equipment is checked against standard equipment or standards that are traceable to national or international standards. Class A Glassware and Glass microliter syringes should be routinely inspected for chips, acid etching or deformity (e.g., bent needle). If the Class A glassware or syringe is suspect, the accuracy of the glassware will be assessed prior to use.

### **21.2 NIST-Traceable Weights and Thermometers**

Reference standards of measurement shall be used for calibration only and for no other purpose, unless it can be shown that their performance as reference standards would not be invalidated.

For NIST-traceable weights and thermometers, the laboratory requires that all calibrations be conducted by a calibration laboratory accredited by A2LA, NVLAP (National Voluntary Laboratory Accreditation Program), APLAC (Asia-Pacific Laboratory Accreditation Cooperation), or EA (European Cooperation for Accreditation). A certificate and scope of accreditation is kept on file at the laboratory.

### **21.3 Reference Standards / Materials**

Reference standards/materials, where commercially available, are traceable to certified reference materials. Commercially prepared standard materials are purchased from vendors accredited by A2LA, NVLAP, or other ISO 17025 accreditation with an accompanying Certificate of Analysis that documents the standard purity. If a standard cannot be purchased from a vendor that supplies a Certificate of Analysis, the purity of the standard is documented by analysis. The receipt of all reference standards must be documented. Reference standards are labeled with a unique Standard Identification Number as assigned in Element 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. This standard is known as the Initial Calibration Verification (ICV) or Quality Control Standard (QCS). In cases where a second standard manufacturer is not available, a vendor certified different lot is acceptable for use as a second source. For unique situations, such as air analysis where no

other source or lot is available, a standard made by a different analyst would be considered a second source. The appropriate Quality Control (QC) criteria for specific standards are defined in laboratory SOPs. In most cases, the analysis of an ICV/QCS 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. For safety requirements, please refer to laboratory method SOPs, the Corporate Environmental Health and Safety Manual (EHSM) and/or the facility EHSM addendum.

Standards and reference materials shall not be used after their expiration dates unless their reliability is verified by the laboratory and their use is approved by the Quality Assurance Manager. The laboratory must have documented contingency procedures for re-verifying expired standards.

#### **21.4 Documentation and Labeling of Standards, Reagents, and Reference Materials**

Reagents must be at a minimum the purity required in the test method. The date of reagent receipt and the expiration date are documented. The lots for most of the common solvents and acids are tested for acceptability prior to company wide purchase. (Refer to TestAmerica's Corporate SOP CA-Q-S-001, Solvent and Acid Lot Testing and Approval.)

All manufacturer or vendor supplied Certificates of Analysis or Purity must be retained, stored appropriately, and readily available for use and inspection. These records are maintained in Element LIMS. Records must be kept of the date of receipt and date of expiration of standards, reagents and reference materials. In addition, records of preparation of laboratory standards, reagents, and reference materials must be retained, stored appropriately, and be readily available for use and inspection. For detailed information on receipt, documentation and labeling of laboratory standards, reagents, and reference materials; please refer to SOPs PE-QAD-012 Receipt Process for General Supplies and Chemicals and PE-QAD-013 Reagent and Standard Preparation, Control and Documentation.

Commercial materials purchased for preparation of calibration solutions, spike solutions, etc., are usually accompanied with an assay certificate or the purity is noted on the label. If the assay purity is 96% or better, the weight provided by the vendor may be used without correction. If the assay purity is less than 96% a correction will be made to concentrations applied to solutions prepared from the stock commercial material.

**21.4.1** All standards, reagents, and reference materials must be labeled in an unambiguous manner. Standards are logged into the laboratory's LIMS system, and are assigned a unique identification number. The following information is typically recorded in the electronic database within the LIMS.

- Standard ID
- Description of Standard
- Department

- Preparer's name
- Final volume and number of vials prepared
- Solvent type and lot number
- Preparation Date
- Expiration Date
- Standard source type (stock or daughter)
- Standard type (spike, surrogate, other)
- Parent standard ID (if applicable)
- Parent Standard Analyte Concentration (if applicable)
- Parent Standard Amount used (if applicable)
- Component Analytes
- Final concentration of each analyte
- Comment box (text field)

Records are maintained electronically for standard and reference material preparation. These records show the traceability to purchased stocks or neat compounds. These records also include method of preparation, date of preparation, expiration date and preparer's name or initials. Preparation procedures are provided in the Method SOPs.

**21.4.2** All standards, reagents, and reference materials must be clearly labeled with a minimum of the following information:

- Expiration Date
- Standard ID (generated from LIMS)
- Special Health/Safety warnings if applicable

Records must also be maintained of the date of receipt for commercially purchased items or date of preparation for laboratory prepared items. Special Health/Safety warnings must also be available to the analyst. This information is maintained as part of the applicable method SOP.

**21.4.3** In addition, the following information may be helpful:

- Date of receipt for commercially purchased items or date of preparation for laboratory prepared items
- Date opened (for multi-use containers, if applicable)
- Description of standard (if different from manufacturer's label or if standard was prepared in the laboratory)
- Recommended Storage Conditions
- Concentration (if applicable)
- Initials of analyst preparing standard or opening container

All containers of prepared reagents must include an expiration date and an ID number to trace back to preparation.

Procedures for preparation of reagents can be found in the Method SOPs.

Standard ID numbers must be traceable through associated logbooks, worksheets and raw data.

All reagents and standards must be stored in accordance to the following priority: 1) with the manufacturer's recommendations; 2) with requirements in the specific analytical methods as specified in the laboratory SOP.

## **SECTION 22. SAMPLING**

### **22.1 Overview**

The laboratory provides sampling services. Sampling procedures are described in SOP PE-SMP-004 Field Sampling for:

- Groundwater Sampling
- Wastewater Sampling
- Soil Sampling

### **22.2 Sampling Containers**

The laboratory offers clean sampling containers for use by clients. These containers are obtained from reputable container manufacturers and meet EPA specifications as required. Any certificates of cleanliness that are provided by the supplier are maintained at the laboratory. Additional information is available in SOP PE-SMP-005 Bottle Preparation.

For Industrial Hygiene, at the client's request, sample media and sampling instructions can be provided. Sample media is shipped to the client via either TestAmerica's courier service or a commercial courier service. Sampling instructions, if requested, are shipped to the client with the sample media.

#### **22.2.1 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:

- Ammonium Chloride – ACS Grade or equivalent
- Ascorbic Acid – ACS Grade or equivalent
- Hydrochloric Acid – Reagent ACS (Certified VOA Free) or equivalent
- MCAA (Chloroacetic Acid) = ACS Grade or equivalent
- Methanol – Purge and Trap grade
- Nitric Acid – Intra-Analyzed or equivalent

- Sodium Hydroxide – Intra-Analyzed or equivalent
- Sulfuric Acid – Intra-Analyzed or equivalent
- Sodium Sulfite – ACS Grade or equivalent
- Sodium Thiosulfate – ACS Grade or equivalent
- Zinc Acetate – ACS Grade or equivalent

### **22.2.2 Industrial Hygiene Sampling Equipment**

In addition to providing clients with sample media and sampling instructions, the laboratory offers sampling equipment for client loan or rental. Loan and rental equipment includes air sampling pumps, impingers, and cyclones. The air sampling pumps are calibrated according to the procedures outlined in SOP PE-SMP-007 – Calibrating Sampling Pumps.

### **22.3 Definition of Holding Time**

The date and time of sampling documented on the COC form will be used to establish the zero (start) date and time at which point the holding time commences. 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 each calendar day measured. Holding times expressed in “hours” (e.g., 6 hours, 24 hours, etc.) are measured from the zero date and time listed on the COC. The first day of holding time ends twenty-four hours after sampling. Holding times for analysis include any necessary reanalysis. However there are some programs that determine holding time compliance based on the date and specific time of analysis compared to the time of sampling regardless of how long the holding time is.

**22.3.1 Semi-Volatiles** - Holding times for sample preparation for semi-volatile organics are measured from the sampling date (and time where applicable) until the day of (and time where applicable) extraction. 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 instrument.

**22.3.2 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 Instrument. The data systems record the start of the analytical run. Extractions, e.g., for high-level soils, must be completed in time to allow for analysis to be initiated within the maximum allowable holding time. Holding time is regulatory program driven.

**22.3.3 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).

### **22.4 Sampling Containers, Preservation Requirements, Holding Times**

The preservation and holding time criteria specified in the laboratory SOPs are derived from the source documents for the methods. General Criteria is specified in Tables 22-1 and 22-2 are. If method required holding times or preservation requirements are not met, the reports will be

qualified using a flag, footnote or case narrative. As soon as possible or "ASAP" is an EPA designation for tests for which rapid analysis is advised, but for which neither EPA nor the laboratory have a basis for a holding time.

## **22.5 Sample Aliquots / Subsampling**

Taking a representative sub-sample from a container is necessary to ensure that the analytical results are representative of the sample collected in the field. The size of the sample container, the quantity of sample fitted within the container, and the homogeneity of the sample need consideration when sub-sampling for sample preparation. It is the laboratory's responsibility to take a representative sub-sample or aliquot of the sample provided for analysis.

Analysts should handle each sample as if it is potentially dangerous. At a minimum, safety glasses, gloves, and lab coats must be worn when preparing aliquots for analysis.

Guidelines on taking sample aliquots & subsampling are located in SOP PE-QAD-003 Sub-sampling.

Table 22-1.

## General Holding Times, Preservation and Container Requirements

<i>P</i> -Poly <i>G</i> -Glass <i>AG</i> -Amber Glass <i>E</i> -Encore <i>TC</i> -Terracore* <i>TLC</i> Teflon®-lined cap <i>TLS</i> Teflon®-lined septum <i>PTFE</i> Fluoropolymer Resin / Teflon® <i>WMG</i> Wide-Mouth Glass <i>ZHS</i> Zero HeadSpace <i>MK</i> Methanol Kit <i>W</i> Water <i>S</i> Soil						
Analysis	Matrix	Method(s)	Recommended Quantity	Preservation	Minimum Volume / Size	Holding Time
<b>Volatile Organics <sup>1</sup></b>						
Dissolved Gasses	<b>W</b>	RSK-175	<sup>2</sup> 3 x 40 ml G-TLS, ZHS	<sup>3</sup> Cool ≤6°C, HCl	1 x 40 ml	14 days
EDB, DBCP & 1,2,3-Trichloropropane	<b>W</b>	EPA 504.1 / SW 8011	3 x 40 ml AG-TLS, ZHS	<sup>4</sup> Cool ≤6°C, HCl	1 x 40 ml	14 days
	<b>S</b>	SW 8011	2-oz.jar	Cool ≤6°C, HCl	10 g	48 hours -14 days <sup>8</sup>
Acrolein, Acrylonitrile & 2-Chloroethyl Vinyl Ether	<b>W</b>	EPA 624 / SW 8260 (5030)	3 x 40 ml G-TLS	Cool ≤6°C	1 x 40 ml	8260 7 days ( <i>unpreserved</i> ) 624 – 72 hrs ( <i>unpreserved</i> )
	<b>S</b>	EPA 8260 (5030/5035)	2-oz. jar <sup>7</sup> 3 x 5g E, 2 x MK or TC	Cool ≤6°C	10 g 1 E	7 days
<sup>5</sup> Gasoline Range Organics (GRO)	<b>W</b>	SW 8015 (5030)	3 x 40 ml G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 ml	14 days
	<b>S</b>	SW 8015 (Low-Level 5030; High-Level 5035)	2-oz. jar <sup>7</sup> 3 x 5g E, 2 x MK or TC	Cool ≤6°C <sup>6</sup> Frozen w/in 48 hours	10 g 1 E	48 hours -14 days <sup>8</sup>
Hydrocarbons (C6-C10)	<b>S</b>	Arizona 8015AZ	4-oz. jar	Cool ≤6°C	30 g	48 hours -14 days <sup>8</sup>
Purgeable Halocarbons	<b>W</b>	EPA 601 / SW 8021, 8015 (5030)	3 x 40 ml G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 ml	14 days ( <i>preserved</i> ) 7 days ( <i>unpreserved</i> )
	<b>S</b>	SW 8021 / 8015 (5035)	<sup>7</sup> 3 x 5g E, 2 x MK or TC	Cool ≤6°C	10 g 1 E	48 hours -14 days <sup>8</sup>
Purgeable Aromatics	<b>W</b>	EPA 602 / SW 8021, 8015 (5030)	3 x 40 ml G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 ml	14 days ( <i>preserved</i> ) 7 days ( <i>unpreserved</i> )
	<b>S</b>	SW 8021 / 8015 (5030 / 5035)	2-oz. jar <sup>7</sup> 3 x 5g E, 2 x MK or TC	Cool ≤6°C	10 g 1 E	48 hours -14 days <sup>8</sup>
TPH by GC/MS	<b>W</b>	SW 8260 Mod. (5030)	3 x 40 ml G-TLS, ZHS	Cool ≤6°C, HCl	1 x 40 ml	14 days
	<b>S</b>	SW 8260 Mod. (5030/5035)	2-oz. jar <sup>7</sup> 3 x 5g E, 2 x MK or TC	Cool ≤6°C <sup>7</sup> Frozen w/in 48 hours	10 g 1 E	48 hours -14 days <sup>8</sup>
<sup>8</sup> Volatile Organics by GC/MS	<b>W</b>	EPA 624 / SW 8260 (5030)	3 x 40 ml G-TLS	Cool ≤6°C, HCl	1 x 40 ml	14 days ( <i>preserved</i> ) 7 days ( <i>unpreserved</i> )
	<b>S</b>	EPA 8260 (5030/5035)	2-oz. jar <sup>7</sup> 3 x 5g E, 2 x MK or TC	<sup>7</sup> Frozen w/in 48 hours	10 g 1 E	48 hours -14 days <sup>8</sup>

Semi-Volatile Organics						
Chlorinated Herbicides	<b>W</b>	EPA 615 / SW 8151	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8151	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
<sup>5</sup> Diesel or Oil Range Organics (DRO/ORO)	<b>W</b>	SW 8015	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8015	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Hydrocarbons (C10-C32)	<b>S</b>	Arizona 8015AZ	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days
Dioxin/Furans	<b>W</b>	EPA 1613 / SW 8280, 8290	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8280, 8290	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Formaldehydes / Acetaldehydes	<b>W</b>	SW 8315 A	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8315 A	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Nitroaromatics/Nitramines (Explosives)	<b>W</b>	SW 8321, 8330, 8332	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8321, 8330, 8332	2-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Oil & Grease	<b>W</b>	EPA 1664A; SM 5520B & C	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$ , HCl	1L	28 days
	<b>S</b>	SW 9071B Mod.	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	28 days
Organophosphorus Pesticides	<b>W</b>	EPA 614 / SW 8141	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8141	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Pesticides and/or PCBs	<b>W</b>	EPA 608 / SW 8081 or 8082	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8081 or 8082	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	60 g	14 days to extract 40 days to analyze
Polynuclear Aromatics	<b>W</b>	EPA 610 / SW 8310, 8270	2 x 1L-AG or WMG TLC	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract 40 days to analyze
	<b>S</b>	SW 8310, 8270	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	14 days to extract 40 days to analyze
Semi-Volatile Organics	<b>W</b>	EPA 625 / SW 8270	2 x 1L-AG or WMG	Cool $\leq 6^{\circ}\text{C}$	1L	7 days to extract

	<b>S</b>	SW 8270	TLC 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	30 g	40 days to analyze 14 days to extract 40 days to analyze
<b>Air Samples</b>						
Volatile Organics	<b>A</b>	EPA 8260	Entech or Summa type Canister	None	6L OR 1L	30 days
Volatile Organics	<b>A</b>	EPA 8021B/8260B	Tedlar Bag	None	1 L	72 hrs <sup>14, 15</sup>
Organochlorine Pesticides	<b>A</b>	TO-10A	PUF Tube, 76 mm	4°C	1 TUBE	7 Days
<b>General Chemistry</b>						
Acidity	<b>W / S</b>	EPA 305.1 / SM 2310B	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	14 days <sup>10</sup>
Alkalinity	<b>W / S</b>	EPA 310.1, 310.2 / SM2320B	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	14 days <sup>10</sup>
Ammonia (as N)	<b>W / S</b>	EPA 350.1, 350.3 / Lachat 10-107-06- 1-B SM 4500-NH3 C, D, E, F, G, H	1 L, P / 8-oz jar	Cool $\leq 6^{\circ}\text{C}$ , H <sub>2</sub> SO <sub>4</sub> to pH <2	100 ml / 100 g	28 days <sup>10</sup>
Anions by IC: Cl, F, Br, SO <sub>4</sub>	<b>W / S</b>	EPA 300.0 / SM 4110B / SW 9056	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 g / 50 ml	28 days
Biochemical Oxygen Demand (BOD)	<b>W</b>	EPA 405.1 / SM 5210B	1L, P	Cool $\leq 6^{\circ}\text{C}$	370 ml	48 hours
Carbon Dioxide	<b>W</b>	SM4500-CO2 C	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	Immediate <sup>11</sup>
Chemical Oxygen Demand (COD)	<b>W / S</b>	EPA 410.1, 410.4 / SM5220 C / HACH 8000	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$ , H <sub>2</sub> SO <sub>4</sub> to pH <2	100 ml / 25 g	28 days <sup>10</sup>
Chloride	<b>W / S</b>	EPA 300.0, 325.2 / SM 4110B, 4500-Cl B, C, D, E / SW 9056, 9251, 9253	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	28 days <sup>10</sup>
Chlorine, Residual	<b>W</b>	EPA 330.4, 330.5 / SM 4500-Cl F, G HACH 8167	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	Immediate <sup>11</sup>

Chromium VI	<b>W / S</b>	EPA 218.4, 218.6 / SW 7196, 7199 / SM 3500-Cr B, D	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	24 hours <sup>10</sup> unpreserved, 28 days preserved <sup>13</sup> (Aqueous);
	<b>S</b>	EPA 6800	4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	25 g	28 days extract, 7 days to analysis (optional 96 hrs. from ext. to analysis in alkaline state)
Color	<b>W</b>	EPA 110.1, 110.2, 110.3 / SM 2120 B, E	500 ml P	Cool $\leq 6^{\circ}\text{C}$	100 ml	48 hours
Conductivity	<b>W</b>	SM 2510B / EPA 120.1 / SW 9050	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	28 days
Cyanide, Amenable or Total	<b>W / S</b>	SM 4500-CN C, E, G / EPA 335.1, 335.3, 335.4 / W 9010, 9012, 9013, 9014 / Lachat 10-201-00-1-A EPA OIA-1677	1L, P / 4-oz. jar (Note: NPDES may require field preservation kit.)	Cool $\leq 6^{\circ}\text{C}$ , NaOH pH > 12; Cool $\leq 6^{\circ}\text{C}$	100 ml / 5 g	14 days <sup>10</sup>
Cyanide, Available	<b>W / S</b>	EPA OIA-1677	Contact the TestAmerica laboratory performing the analysis for method-exclusive containers			14 days
Flashpoint / Ignitability	<b>W / S</b>	SW 1010, 1020, 1030	250 ml, G / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 50 g	28 days
Fluoride	<b>W / S</b>	EPA 300.0, 340.2 / SW 9056, 9214 / SM 4110B, 4500-F C	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	28 days <sup>10</sup>
Hardness	<b>W</b>	EPA 130.2 / SM 2340C	500 ml, P	Cool $4^{\circ}\text{C}$ , HNO <sub>3</sub> pH < 2	100 ml	180 days
MBAS (Surfactants)	<b>W</b>	SM 5540C	1L, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	48 hours
Nitrate	<b>W / S</b>	EPA 300.0, 353.2 / SM 4110B, SM 4500-NO3 D, E, F, H / SW 9056	1L, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	100 ml / 25 g	48 hours <sup>10</sup>

Nitrate + Nitrite	<b>W / S</b>	EPA 300.0, 353.2 / SW 9056 SM 4110B, 4500-NO <sub>3</sub> D, E, F, H Lachat 10-107-04-1-C	1L, P / 4-oz. jar	Cool ≤6°C, H <sub>2</sub> SO <sub>4</sub> to pH < 2; Cool ≤6°C	200 ml / 25 g	28 days <sup>10</sup>
Nitrite	<b>W / S</b>	EPA 300.0, 354.1 / SM 4110B, 4500-NO <sub>2</sub> B / SW 9056 / HACH 8507	1L, P / 4-oz. jar	Cool ≤6°C	100 ml / 25 g	48 hours <sup>10</sup>
Nitrogen, Kjeldahl (TKN)	<b>W / S</b>	SM 4500 NorgC / EPA 351.2, 351.3, 351.4 Lachat 10-107-06-2-E	1L, P / 4-oz. jar	Cool ≤6°C, H <sub>2</sub> SO <sub>4</sub> pH < 2; Cool ≤6°C	100 ml / 25 g	28 days <sup>10</sup>
Odor	<b>W</b>	EPA 140.1 / SM 2150B	500 ml, G	Cool ≤6°C	100 ml	24 hours
Oxygen, Dissolved (DO)	<b>W</b>	EPA 360.1, 360.2 / SM 4500-O G	2 x 40 ml-VOA; 250 ml P	Cool ≤6°C, no HS	100 ml	Immediate <sup>11</sup>
Perchlorate	<b>W / S</b>	EPA 314.0	500 ml, P / 4-oz. jar	Cool ≤6°C	100 g / 50 ml	28 days
Perchlorate	<b>W</b>	EPA 331 / SW 6850, 6860, 8321	500 ml, P	Cool ≤6°C, Sterile	50 ml	28 days
pH	<b>W / S</b>	EPA 150.1 / SM4500-H B / SW 9040, 9045	500 ml, P, / 4-oz. jar	Cool ≤6°C	100 ml / 5 g	Immediate <sup>11</sup>
Phenols, Total	<b>W / S</b>	EPA 420.1, 420.2, 420.4 / SW 9065, 9066	1L, AG / 4-oz. jar	Cool ≤6°C, H <sub>2</sub> SO <sub>4</sub> pH < 2; Cool ≤6°C	100 ml / 5 g	28 days
Phosphate, Ortho	<b>W / S</b>	EPA 300.0, 365.1, 365.2, 365.3, 365.4 / SM 4110B, 4500-P E / SW 9056	500 ml, P / 4-oz. jar	Cool ≤6°C	5 g / 50 ml	48 hours
Phosphorus (ICP)	<b>W / S</b>	EPA 200.7 / SW 6010	500 ml, P / 4-oz. jar	Cool ≤6°C, HNO <sub>3</sub> pH <2;	100 ml / 10 g	180 days

				Cool $\leq 6^{\circ}\text{C}$		
Phosphorus (Gen Chem)	<b>W / S</b>	EPA 365.1, 365.2, 365.3, 365.4 / SM 4500-P B, E, F	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$ , H <sub>2</sub> SO <sub>4</sub> pH < 2; Cool $\leq 6^{\circ}\text{C}$	100 ml / 5 g	28 days
Silica	<b>W</b>	EPA 200.7 / SM 4500-SiO <sub>2</sub> C, D; SM 3120B	500 ml P, PTFE, Quartz	Cool $\leq 6^{\circ}\text{C}$	100 ml	28 days
Solids, Settleable	<b>W</b>	EPA 160.5 / SM 2540F	1L, P	Cool $\leq 6^{\circ}\text{C}$	1000 ml	48 hours
Solids, Total Dissolved (TDS)	<b>W</b>	EPA 160.1 / SM 2540C	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	7 days
Solids, Total Suspended (TSS)	<b>W</b>	EPA 160.2 / SM 2540D / USGS I-3765-85	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 ml	7 days
Solids, Total Volatile (TVS)	<b>W</b>	EPA 160.4	500 ml, P	Cool $\leq 6^{\circ}\text{C}$	100 mL	7 days
Solids, Total (TS)	<b>W</b>	SM 2540B	1 L, P	Cool $\leq 6^{\circ}\text{C}$	500 mL	7 days
Sulfate	<b>W / S</b>	EPA 300.0, 375.4 / SM 4110B, 4500-SO <sub>4</sub> E, 426 C, 15th Ed. / SW 9038, 9056 / ASTM D516-90,02	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$	50 ml / 50 g	28 days <sup>10</sup>
Sulfide, Total (TS)	<b>W / S</b>	EPA 376.1, 376.2 / SM 4500-S-2 D, E, F / SW 9030, 9034 / HACH 8216	500 ml, P / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$ , NaOH+Zn Acetate pH >9; Cool $\leq 6^{\circ}\text{C}$	100 ml / 50 g	7 days
Total Organic Carbon (TOC)	<b>W / S</b>	EPA 415.1, 415.2 / SM 5310 B, C / SW 9060 / Lloyd Kahn	250 ml, AG or 2 x 40 ml G-TLS, ZHS 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$ , H <sub>2</sub> SO <sub>4</sub> or HCl pH < 2; Cool $\leq 6^{\circ}\text{C}$	100 ml / 5 g	28 days 14 days (Lloyd Khan)
	<b>S</b>	Walkley Black	4-oz. jar w/PFTE-lined lid	Cool $\leq 6^{\circ}\text{C}$	50 g	28 days
Total Organic Halides (TOX)	<b>W / S</b>	EPA 450.1 / SM 5350B / SW 9020	500 ml AG-TLC / 4-oz. jar	Cool $\leq 6^{\circ}\text{C}$ , H <sub>2</sub> SO <sub>4</sub> pH <	100 ml / 50 g	28 days

				2; Cool ≤6°C		
Total Petroleum Hydrocarbon (TPH)	<b>W</b>	EPA 1664 (SGT HEM)	2 x 1 L- AG, TLC	Cool ≤6°C, H <sub>2</sub> SO <sub>4</sub> pH < 2;	100 m	28 days
	<b>S</b>	SW 9071B Mod.	4-oz. jar	Cool ≤6°C	50 g	28 days
Turbidity	<b>W</b>	EPA180.1 / SM 2130B	500 ml, P	Cool ≤6°C	50 ml	48 hours
<b>Metals</b>						
Cation Exchange Capacity (CEC)	<b>S</b>	SW 9081	4-oz. jar	Cool ≤6°C	50 g	28 days
Chromium VI	<b>W</b>	EPA 218.6 / SW 7199, 7196	500 ml, P	Cool ≤6°C	50 ml	24 hours
	<b>S</b>	SW 7199, 7196A	4-oz. jar	Cool ≤6°C	20 g	30 days
Mercury	<b>W</b>	EPA 245.1, 245.2 / SW 7470	500 ml, P	HNO <sub>3</sub> to pH < 2	100 ml	28 days
	<b>S</b>	EPA 1630, 1631 / SW 7471A	4-oz. jar	Cool ≤6°C	10 g	28 days
Metals, Dissolved (Field Filtered)	<b>W</b>	EPA 200.7, 200.8 / SW 6010, 6020	500 ml, P	HNO <sub>3</sub> to pH < 2	100 ml	180 days
Metals, Total	<b>W</b>	EPA 200.7, 200.8 / SW 6010, 6020	500 ml, P	HNO <sub>3</sub> to pH < 2	100 ml	180 days
	<b>S</b>	SW 6010, 6020	4-oz. jar	Cool ≤6°C	5 g	180 days
Organic Lead by GFAA	<b>W</b>	HML 939-M	1L AG, ZHS	Cool ≤6°C	200 ml	14 days
	<b>S</b>	HML 939-M	4-oz. jar	Cool ≤6°C	50 g	14 days
TCLP, STLC, SPLP metals	<b>W</b>	40 CFR Part 136, Md. 1311, 1312	4 x 40 ml G-TLS, ZHS 4 x 1 L, G 1 x 500 ml, P	Cool ≤6°C	100g / 50g / 100g	180 days <sup>12</sup>
	<b>S</b>	40 CFR Part 136, Md. 1311, 1312	8-oz. jar	Cool ≤6°C	100g / 50g / 100g	180 days <sup>12</sup>
<b>Microbiology</b>						
Chlorophyll a	<b>W</b>	SM 10200 H	1 L, AP or AG foil wrapped	Cool ≤6°C	100 ml	48 hours
Coliform, Total	<b>W</b>	SM 9222	250 ml P,G	Cool 10°C,	100 ml	6 hours;

			(sterile)	Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>		30 hours (MA)
Coliform, Fecal	<b>W</b>	SM 9221, 9222	250 ml P,G (sterile)	Cool 10°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	100 ml	6 hours
E. Coli	<b>W</b>	SM 9221, 9223 / IDEXX / Colisure	250 ml P,G (sterile)	Cool 10°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	100 ml	6 hours; 30 hours (MA)
Enterococci	<b>W</b>	SM 9230 / ENTEROLERT / ASTM D6503-99	250 ml P,G (sterile)	Cool 10°C	100 ml	6 hours
Hetrotrophic Plate Count	<b>W / S</b>	SM 9215 / IDEXX	250 ml P,G (sterile)	Cool 10°C, Na <sub>2</sub> S <sub>2</sub> O <sub>3</sub>	100 ml	8 hours
<b>Radiochemistry</b>						
Carbon-14	<b>W</b>	EERF C-01-1	100 mL	none	75 mL	6 months
	<b>S</b>	EERF C-01-1	5 grams, P	none	1 gram	6 months
Cesium-134	<b>W</b>	EPA 901; DOE GA- 01-R	1 L, P	None	1 L	6 months
	<b>S</b>	EPA 901; DOE GA- 01-R	650 grams, P	None	650 grams	6 months
Gross Alpha/Beta	<b>W</b>	EPA 900 & SW 9000 Series	1 L, P	HNO <sub>3</sub> pH < 2	500 ml	6 months
	<b>S</b>	EPA 900 & SW 9000 Series	10 grams, P	None	5 grams	6 months
Iodine-129	<b>W</b>	Standard Method 7500-IB	2 L, P	None	2 L	6 months
Iodine-131	<b>W</b>	EPA 902.0, 901.1	1 L, P	None	1 L	16 days
Isotopic Analysis: Am, Cm, Np, Pu, Th, U	<b>W</b>	DOE A-01-R	1 L, P	HNO <sub>3</sub> pH < 2	1 L	6 months
	<b>S</b>	DOE A-01-R	10 grams, P	None	10 gram	6 months
Nickel 59/63; Iron-55	<b>W</b>	Eichrom Technologies Methods	1 L, P	HNO <sub>3</sub> pH < 2	500 mL	6 months
	<b>S</b>	Eichrom Technologies Methods	5 grams, P	None	5 grams	6 months
Plutonium-241	<b>W</b>	Lab SOP/Liquid Scintillation Counting	1 L, P	HNO <sub>3</sub> pH < 2	500 mL	6 months

	<b>S</b>	Lab SOP/Liquid Scintillation Counting	10 grams, P	None	10 gram	6 months
Radium 226/228	<b>W</b>	EPA 903.1, 904.0	1 L, P	HNO <sub>3</sub> pH < 2	1 L	6 months
	<b>S</b>	EPA 903.1, 904.0 SW 846 9315/9320	10 grams, P	None	10 gram	6 months
Radon 222	<b>W</b>	EPA 913	3 x 40 mL	None	40 ml	4 days
Strontium 89, 90	<b>W</b>	EPA 905	1 L, P	HNO <sub>3</sub> pH < 2	1 L	6 months
	<b>S</b>	EPA 905; DOE Sr-03-RC	10 grams, P	None	2 grams	6 months
Technetium-99	<b>W</b>	Eichrom Method TCW01	500 mL	HNO <sub>3</sub> pH < 2	250 mL	6 months
	<b>S</b>	Eichrom Method TCS01	10 grams, P	None	5 grams	6 months
Tritium	<b>W</b>	EPA 906.0	1 L, P	None	100 mL	6 months
	<b>S</b>	EPA 906.0	100 grams, P	None	30 gram	6 months
Uranium-12	<b>W</b>	EPA 908.0, 908.1	2 L, P	HNO <sub>3</sub> pH < 2	2 L	6 months
<b>Bioassay</b>						
Acute-24/48-hr	<b>W</b>	EPA 821-R-02-012	2gal Eff & 3gal River (if required)	Cool ≤6°C	See Quantity	36 hours
Acute-96-hr	<b>W</b>	EPA 821-R-02-012	2gal Eff & 3gal River (if req)	Cool ≤6°C	See Quantity	36 hours
Chronic-7-day	<b>W</b>	EPA 821-R-02-013	2gal Eff & 3gal River (if req) per species x 3days	Cool ≤6°C	See Quantity	36 hours

**Footnotes:**

\* Terracore kits usually include 3-vial kits as (MeOH/H<sub>2</sub>O/H<sub>2</sub>O); 3-vial kits as (MeOH/Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>/Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>); or 4-vial kits (MeOH/MeOH/H<sub>2</sub>O/H<sub>2</sub>O). Some kits come with the % moisture cup/jar, and a disposable t-handle. The kits with the H<sub>2</sub>O do not have the stir-bar.

> CLP Methods are also available - contact a Project Manager for additional information. <

<sup>1</sup> Additional soil volume of 20 grams is required for Total Solids determination. This can be collected in a 2 oz. jar.

<sup>2</sup>Samples for analysis of carbon dioxide should be collected in 40 mL VOA vials without preservative.

<sup>3</sup> Sample temperatures to be maintained at 0-≤6°C.

<sup>4</sup> EPA 504.1: If residual chlorine is expected to be present, samples should be neutralized with Sodium Thiosulfate.

<sup>5</sup> Contact the laboratory for information on other state-specific DRO and GRO methods.

<sup>6</sup> Samples can be frozen within 48-hrs of collection. Preserve prior to analysis. Analyze within 14 days of collection (samples need to be extruded prior to freezing).

<sup>7</sup> Within 48 hours of sample collection, the sample in the EnCore™ sampler must be transferred to the sample vial containing organic-free water and frozen or if required, transferred to vial containing preservative if effervescence test was negative.

<sup>8</sup> Holding times for soil samples are regulatory program specific. Contact the laboratory for additional information.

<sup>9</sup> Samples where vinyl chloride, styrene, or 2-chloroethyl vinyl ether are analytes of interest, collect a second set of samples without acid preservatives and analyze as soon as possible.

NIOSH & OSHA methods under the Industrial Hygiene Program; and EPA methods for the Clean Air Program are also available for the analysis of Volatile Organics.

<sup>10</sup> For a Solid/Waste matrix, some inorganic parameters will undergo a DI water leach prior to analysis.

<sup>11</sup> Immediate equals 15 minutes from sampling or field test

<sup>12</sup> TCLP/STLC/SPLP Hold

Times:

Metals -Liquids:	Collection to extract 180 (28d Hg), prep to analysis 180d (28d Hg)
Semi-volatile - Liquids:	Collection to extract 14d, extract to prep 7d, prep to analysis 40d
Volatiles - Liquids:	Collection to extraction 14d, extraction to analysis 14d
Metals -Solids:	Collection to extract 180 (28d Hg), prep to analysis 180d (28d Hg)
Semi-volatiles - Solids:	Collection to extract 14d, extract to prep 7d, prep to analysis 40d
Volatiles - Solids:	Collection to extraction 14d, extraction to analysis 14d

<sup>13</sup> To achieve the 28-day holding time, use the ammonium sulfate buffer solution specified in EPA Method 218.6

<sup>14</sup> Holding Time is based on SW 846 Method 0040 "SAMPLING OF PRINCIPAL ORGANIC HAZARDOUS CONSTITUENTS FROM COMBUSTION SOURCES USING TEDLAR® BAGS". Some states specifically enforce this holding time (e.g., Florida, New Jersey) and others have not specified this information in their regulatory requirements.

<sup>15</sup> The holding time is 72 hours unless the laboratory has a documented validation study that indicates a longer HT is acceptable for the analytes of interest.

**Additional Comments:**

- a. For samples requiring MS/MSD, collect triple the quantity.
- b. For bacteriological and organic parameters, add sodium thiosulfate if residual chlorine is present.
- c. Trademarks & trade names used in this document are the property of their respective owners.

**Volume Conversion Guide:**

**2 oz. Jar = 50 g**

**4 oz. Jar = 100 g**

**8 oz. Jar = 200 g**

**16 oz. Jar = 400 g**

**Table 22-2. Industrial Hygiene – Sample Receiving Guide**

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Acetaldehyde	OSHA 1007 (Modified)	AT N571 Passive Monitor	0.00977 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Acetone	NIOSH 1300	150-mg Charcoal Tube	0.01 - 0.2	0.5 - 3	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	OSHA 69	225-mg Anasorb CMS Tube	0.05	3	17 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0401	2 Hrs Max.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0152	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00160 (#546) 0.0106 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Acetonitrile	3M (Modified)	3M 3520	0.0482	2 Hrs	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Acetylene	EPA 3C/ASTM D1946	Entech Canister	400 ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1 L	Grab	5 Days	Should be stored at room temperature.
Acrylonitrile	NIOSH 1604	150-mg Charcoal Tube	0.01 - 0.2	3.5 - 20	7 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0438	8 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Aldehydes	EPA TO-11A	DNPH-coated Silica Gel	0.1 - 1.5	1 - 15	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	EPA IP-6A (Modified)	DNPH-coated Silica	0.1 - 1.5	1 - 15	14 Days	Should be shipped on ice or equivalent;

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
		Gel				refrigerate upon receipt.
	EPA IP-6C (Modified)	SKC UME <sub>x</sub> -100 Passive Badge	28.6	15 min. or 8 Hrs	21 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Aluminum	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 960 / 5 - 100	Indefinite	Should be stored at room temperature.
Antimony	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 960 / 50 - 2000	Indefinite	Should be stored at room temperature.
Arsenic	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	5-2000	Indefinite	Should be stored at room temperature.
Barium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 960 / 50-2000	Indefinite	Should be stored at room temperature.
Benzaldehyde	OSHA 1007 (Modified)	Assay N571 Passive Badge	0.00581 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Benzene	NIOSH 1501	150-mg Charcoal Tube	< 0.2	5 - 30	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0355	8 Hrs Max.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0160	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00096 (#546) 0.00785 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Beryllium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	1250 - 2000	Indefinite	Should be stored at room temperature.
Boron	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	25 - 2000	Indefinite	Should be stored at room temperature.
Butane (n-Butane)	ASTM D1945-03	Entech Canister	0.01 - 1	1 L	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	0.01 - 1	1 L	72 Hrs.	Should be stored at room temperature.
1-Butanol (n-butyl alcohol; n-butanol)	NIOSH 1401/1405	150-mg Charcoal Tube	0.01 - 0.2	2 - 10	Not Determined	Should be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0343	8 Hrs.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
2-Butoxyethanol (Butyl Cellosolve)	NIOSH 1403	150-mg Charcoal Tube	0.01 - 0.05	2 - 10	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0282	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
n-Butyl Acetate	NIOSH 1450	150-mg Charcoal Tube	0.01 - 0.2	1 - 10	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0316	8 Hrs.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0132	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00087 (#546) 0.00651 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Butyraldehyde	OHA 1007 (Modified)	Assay N571 Passive Badge	0.00683	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Cadmium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	13 - 2000	Indefinite	Should be stored at room temperature.
Calcium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 960 / 5 - 2000	Indefinite	Should be stored at room temperature.
Carbon Black	NIOSH 5000	37-mm pre-weighed PVC Filter, 5-um pore size	1 - 2	30 - 570	Indefinite	Should be stored at room temperature.
Carbon Dioxide	EPA 3C/ASTM D1946	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1 L	Grab	5 Days	Should be stored at room temperature.
Carbon Monoxide	EPA 3C/ASTM D1946	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1 L	Grab	5 Days	Should be stored at room temperature.
Carbon Tetrachloride	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	3 - 150	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
	3M (Modified)	3M Badge (3500 or 3520)	0.0302	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0145	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00109 (#546) 0.00605 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Chlorobenzene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1.5 - 40	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0293	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0142	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology (Modified)	Assay N546 or N566 Badge	0.00102 (#546) 0.00708 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Chloroform	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1 - 50	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0335	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0130	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology (Modified)	Assay N546 or N566 Badge	0.00146 (#546) 0.00688 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Chromium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 1000	Indefinite	Should be stored at room temperature.
Cobalt	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	25 - 2000	Indefinite	Should be stored at room temperature.
Copper	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 1000	Indefinite	Should be stored at room temperature.
Cresols	NIOSH 2546	150-mg XAD-7	0.01 - 0.1	1 - 24	Undetermined	Should be shipped on ice or equivalent;

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
		Tube			ed	refrigerate upon receipt.
Crotonaldehyde	Assay Technology(Mod)	AT N571 Passive Monitor	0.00716 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Cumene	NIOSH 1501	150-mg Charcoal Tube	< 0.2	1 - 30	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0245	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0128	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology (Modified)	Assay N546 or N566 Badge	0.00083 (#546) 0.00685 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Cyclohexane	NIOSH 1500	150-mg Charcoal Tube	0.01 - 0.2	2.5 - 5	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Cyclohexanone	NIOSH 1300	150-mg Charcoal Tube	0.01 - 0.2	1 - 10	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0289	8 Hrs.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Diborane	NIOSH 6006	PTFE filter + oxidizer impregnated charcoal tube	0.5 - 1.0	60 - 260	7 Days	Should be stored at room temperature.
1,2-Dichlorobenzene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1 - 10	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0278	8 Hrs.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
1,3-Dichlorobenzene	3M (Modified)	3M Badge (3500 or 3520)	0.0267	8 Hrs.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
1,4-Dichlorobenzene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1 - 8	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
	3M (Modified)	3M Badge (3500 or 3520)	0.0278	8 Hrs.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
1,1-Dichloroethane	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	0.5 - 15	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
1,2-Dichloroethane	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1 - 50	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0332	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
cis-1,2-Dichloroethylene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	0.2 - 5	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
trans-1,2-Dichloroethylene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	0.2 - 5	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Diesel Range Hydrocarbons C10 - C22	NIOSH 1550	150-mg Charcoal Tube	0.01 - 0.2	1.3 - 20	14 Days	Should be stored at room temperature.
Diethyl Ether (Ethyl ether, Ethyl oxide)	3M (Modified)	3M 3520 Passive Monitor	0.0368	4 Hr	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
2,5-Dimethylbenzaldehyde	Assay Technology(Mod)	AT N571 Passive Monitor	0.00479 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
1,4-Dioxane	NIOSH 1602	150-mg Charcoal Tube	0.01 - 0.2	0.5 - 15	6 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
n-Dodecane (C12)	3M (Modified)	3M Badge (3500 or 3520)	0.0215	Undetermined	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
Epichlorohydrin	NIOSH 1010	150-mg Charcoal Tube	0.01 - 0.2	2 - 30	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Ethane	ASTM D1945-03	Entech Canister	0.01 - 1	1	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	0.01 - 1	1	72 Hrs	Should be stored at room temperature.
Ethanol	NIOSH 1400	150-mg Charcoal Tube	0.05	0.1 - 10	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M 3520 Passive Monitor	0.0437	1 Hr Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
	SKC (Modified)	SKC 575-002 Passive Badge	0.0209	4 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00154 (#546) 0.0111 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Ethyl Acetate	NIOSH 1457	150-mg Charcoal Tube	0.01 - 0.2	0.1 - 10	6 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0144	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Ethylbenzene	NIOSH 1501	150-mg Charcoal Tube	< 0.2	1 - 24	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0273	8 Hrs. Max	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N545 or N546 Badge	0.00091 (#546) 0.0073 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Fixed Gas Screen (H <sub>2</sub> , C <sub>2</sub> H <sub>2</sub> , CO <sub>2</sub> , CO, CH <sub>4</sub> , N <sub>2</sub> , O <sub>2</sub> )	EPA 3C/ASTM D1946	Entech Canister	400 mL or 1L	Grab or Time Integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1	Grab	5 Days	Should be stored at room temperature.
Formaldehyde	NIOSH 2016 (Modified)	DNPH-coated Silica Gel	0.03 - 1.5	1 - 15	34 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	OSHA 1007 (Modified)	AT N571 Passive Monitor	0.01305 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	OSHA 1007 (Modified)	SKC UME <sub>x</sub> -100 Passive Badge	28.6	15 min. or 8 Hrs	21 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	EPA IP-6C (Modified)	SKC UME <sub>x</sub> -100 Passive Badge	28.6	15 min. or 8 Hrs	21 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Gasoline Range Hydrocarbons C <sub>6</sub> - C <sub>10</sub>	EPA TO-15 (Modified)	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Gasoline Range Hydrocarbons C6 - C10	NIOSH 1550	150-mg Charcoal Tube	0.01 - 0.2	1.3 - 20	14 Days	Should be stored at room temperature.
Glutaraldehyde	OSHA 64 (Modified)	DNPH-coated Glass Fiber Filters	1 - 2	15 - 480	17 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	NIOSH 2532 (Modified)	DNPH-coated Silica Gel	0.05 - 0.5	1 - 30	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology (Modified)	AT N571 Passive Monitor	0.00603 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Heptane (n-Heptane)	NIOSH 1500	150-mg Charcoal Tube	0.01 - 0.2	Undetermined	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Hexaldehyde	OSHA 1007 (Modified)	AT N571 Passive Monitor	0.00540 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
1,6-Hexamethylene Diisocyanate (1,6-HDI)	OSHA 42	37-mm Glass Fiber Filter coated with 1,2PP	1	15	18 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Hexane (n-Hexane)	NIOSH 1500	150-mg Charcoal Tube	0.01 - 0.2	Undetermined	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.032	8 Hrs Max.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0143	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	ASTM D1945-03	Entech Canister	0.01 - 1	1	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	1	Grab	72 Hrs	Should be stored at room temperature.
2-Hexanone	NIOSH 1300	150-mg Charcoal Tube	0.01 - 0.2	1 - 10	7 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Hexavalent Chromium (Soluble)	NIOSH 7600	37-mm PVC Filter, 5-um	1 - 4	100 - 400	14 Days	Should be stored at room temperature.
Hexavalent Chromium	OSHA ID-215	37-mm PVC Filter, 5-um	2	30 - 960	Ship overnight, within	Should be stored at room temperature.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
					24 hours of sampling.	
Hydrobromic Acid	NIOSH 7903	600-mg Cleaned Silica Gel Tube	0.2 - 0.5	3 - 100	21 Days	Should be stored at room temperature.
Hydrochloric Acid	NIOSH 7903	600-mg Cleaned Silica Gel	0.2 - 0.5	3 - 100	21 Days	Should be stored at room temperature.
Hydrofluoric Acid	NIOSH 7903	600-mg Cleaned Silica Gel	0.2 - 0.3	3 - 100	21 Days	Should be stored at room temperature.
Hydrogen	EPA 3C/ASTM D1946	Entech Canister	400 mL or 1L	Grab or Time Integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1	Grab	5 Days	Should be stored at room temperature.
Hydrogen Cyanide	NIOSH 6010	800-mg Soda Lime Tube	0.05 - 0.2	2 - 90	14 Days	Should be stored at room temperature.
Hydrogen Sulfide	NIOSH 6013	600 mg - LOW SO4 Charcoal tube, Orbo 34	0.1 - 1.5	20 - 40	30 Days	Should be stored at room temperature.
Iron	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 960 / 5 - 100	Indefinite	Should be stored at room temperature.
Isopropanol (2-Propanol)	NIOSH 1400	150-mg Charcoal Tube	0.01 - 0.2	0.3 - 3	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M 3520 Passive Monitor	0.0994	8 Hrs	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0178	4 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Isobutyl Acetate	3M (Modified)	3M Badge (3500 or 3520)	0.0310	8 Hrs	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
Isopropyl Acetate	3M (Modified)	3M Badge (3500 or 3520)	0.0317	7 Hrs	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
Isovaleraldehyde	OSHA 1007 (Modified)	AT N571 Passive Monitor	0.00601 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Lead	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	50 - 2000	Indefinite	Should be stored at room temperature.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Lithium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	100 - 2000	Indefinite	Should be stored at room temperature.
Magnesium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 960 / 5 - 67	Indefinite	Should be stored at room temperature.
Manganese	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 960 / 5 - 200	Indefinite	Should be stored at room temperature.
Mercury	NIOSH 6009	200-mg Anasorb C300 Tube	0.15 - 0.25	2 - 100	30 Days	Should be stored at room temperature.
Mercury, Inorganic	OSHA ID-140	800-mg Anasorb C300 Cartridge	0.02	9.6	30 Days	Should be stored at room temperature.
Methane	EPA 3C/ASTM D1946	Entech Canister	Grab or Time integrated	400 mL or 1L	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	Grab	1 L	5 Days	Should be stored at room temperature.
	ASTM D1945-03	Entech Canister	Grab or Time Integrated	400 mL or 1 L	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	Grab	1	72 Hrs	Should be stored at room temperature.
Methanol	NIOSH 2000	150-mg Silica Gel Tube	0.02 - 0.2	1 - 5	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
4-4'-Methylene Bisphenyl Isocyanate (4,4'-MDI)	OSHA 47	37-mm Glass Fiber Filter coated with 1,2PP	1 L/min	15	15 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Methylal	NIOSH 1611	150-mg Charcoal Tube	0.1-0.2	1-3	Unknown	Should be shipped on ice or equivalent; refrigerate upon receipt.
Methylene Chloride	NIOSH 1005	2 Charcoal Tubes in series, 150-mg each	0.01 - 0.2	0.5 - 2.5	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M 3520 Passive Monitor	0.03\$79*	6 Hrs Max.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
Methyl Ethyl Ketone	OSHA 1004	225-mg Anasorb CMS Tube	0.05	≤ 12	15 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500)	0.0363	8 Hrs Max.	3 Weeks	May be shipped on ice or equivalent;

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
		or 3520)				refrigerate upon receipt.
	SKC + OSHA 1004	SKC 575-002 Passive Badge	0.01688	8 Hrs Max.	25 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Methyl Isobutyl Ketone	OSHA 1004	225-mg Anasorb CMS Tube	0.05	≤ 12	15 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC + OSHA 1004	SKC 575-002 Passive Badge	0.01362	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	NIOSH 1300	150-mg Charcoal Tube	1 - 10	0.01 - 0.2	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0300	8 Hrs	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00092 (#546) 0.00751 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Methyl Methacrylate	3M (Modified)	3M Badge (3500 or 3520)	0.0318	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0131	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00100 (#546) 0.00751 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Methyl tert-Butyl Ether (MTBE)	NIOSH 1615	150-mg Charcoal Tube	0.1 - 0.2	2 - 96	5 Days / 3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0308	8 Hrs.	28 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Molybdenum	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 960 / 5 - 67	Indefinite	Should be stored at room temperature.
Naphthas (client must submit bulk liquid sample to be used as reference)	NIOSH 1550	150-mg Charcoal Tube	0.01 - 0.2	1.3 - 20	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
standard)						
Natural Gas Screen (CH <sub>4</sub> , C <sub>2</sub> H <sub>6</sub> , C <sub>3</sub> H <sub>8</sub> , C <sub>4</sub> H <sub>10</sub> , C <sub>5</sub> H <sub>12</sub> , C <sub>6</sub> H <sub>14</sub> )	ASTM D1945-03	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	Grab	1	72 Hrs.	Should be stored at room temperature.
Nickel	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 1000	Indefinite	Should be stored at room temperature.
Nicotine	NIOSH 2551	120-mg XAD-4 Tube	0.1 - 1	0.5 - 600	14 Days - Dark	Should be shipped on ice or equivalent; refrigerate upon receipt. Avoid Light exposure.
Nitric Acid	NIOSH 7903	600-mg Cleaned Silica Gel Tube	0.2 - 0.5	3 - 100	21 Days	Should be stored at room temperature.
Nitrogen	EPA 3C/ASTM D1946	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1 L	Grab	5 Days	Should be stored at room temperature.
Octane (n-Octane)	NIOSH 1500	150-mg Charcoal Tube	0.01 - 0.2	4	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0266	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0127	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00078 (#546) 0.00703 (#546)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Oxygen	EPA 3C/ASTM D1946	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	EPA 3C/ASTM D1946	Flex Foil Bag	1 L	Grab	5 Days	Should be stored at room temperature.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Particulates, Respirable Dusts	NIOSH 0600	37-mm pre-weighed PVC Filter, 5-um pore size, w/al cyclone	2.5	20 - 400	Indefinite	Should be stored at room temperature.
Particulates, Total Dusts	NIOSH 0500	37-mm pre-weighed PVC Filter, 5-um pore size	1 - 2	7 - 133	Indefinite	Should be stored at room temperature.
Pentane (n-Pentane)	NIOSH 1500	150-mg Charcoal Tube	0.01 - 0.4	4	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M 3520 Passive Monitor	0.0353	3 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Monitor	0.0149	8 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology (Modified)	Assay N546 or N566 Badge	0.00105 (#546) 0.00886 (#566)	8 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	ASTM D1945-03	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	1 L	Grab	72 Hrs	Should be stored at room temperature.
Pesticides, Organochlorine	TO-10A	PUF Tube, 76 mm	1 - 5	240 - 7200	10 Days	Must be shipped on wet ice; refrigerate upon receipt.
Pesticides, Organophosphorus	NIOSH 5600	OVS-2 Tube; 13-mm Quartz Filter with 450-mg XAD-2	0.2 - 1	12 - 240	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Pesticides, Organophosphorus	TO-10A	PUF Tube, 76 mm	1 - 5	240 - 7200	10 days	Must be shipped on wet ice; refrigerate upon receipt.
Phenol	NIOSH 2546	150-mg XAD-7 Tube	0.01 - 0.1	1 - 24	Undetermined	Should be shipped on ice or equivalent; refrigerate upon receipt.
4-Phenylcyclohexene	OSHA In-House Method (Modified)	150 or 600 mg Charcoal Tube	0.2	10 - 360	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Phosphine	OSHA 1003	37-mm glass fiber filter with a mercuric chloride treated polyester filter	TWA: 1.0 STEL: 2.0	TWA: 240 L max STEL: 30 L max	17 Days (filter extremely short holdtime)	Should be shipped on ice or equivalent; refrigerate upon receipt.
Phosphoric Acid	NIOSH 7903	600-mg Cleaned Silica Gel	0.2 - 0.5	3 - 100	21 Days	Should be stored at room temperature.
Polychlorinated Biphenyls (PCBs) - Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260.	NIOSH 5503	13-mm, Glass fiber filter in series with a 150-mg Florisil	0.05 - 0.2	1 - 50	2 Months for Tubes	Should be shipped on ice or equivalent; refrigerate upon receipt.
Polychlorinated Biphenyls (PCBs) - Aroclors 1016, 1221, 1232, 1242, 1248, 1254, and 1260.	TO-10A	PUF Tube, 76 mm	1 - 5	240 - 7200	10 Days	Must be shipped on wet ice; refrigerate upon receipt.
Polynuclear Aromatic Hydrocarbons (PAH/PNA)	NIOSH 5506	37-mm, 2-um PTFE filter in series with a 120-mg XAD-2	2	200 - 1000	Unknown-Protect from light/heat	Should be shipped on ice or equivalent; refrigerate upon receipt.
Potassium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 960 / 5 - 1000	Indefinite	Should be stored at room temperature.
Propane	ASTM D1945-03	Entech Canister	400ml or 1 L	Grab or Time integrated	28 Days	Should be stored at room temperature.
	ASTM D1945-03	Tedlar Bag	1 L	Grab	72 Hrs.	Should be stored at room temperature.
1-Propanol	NIOSH 1401/1405	150-mg Charcoal Tube	0.01-0.2	1-10	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Propionaldehyde	OSHA 1007 (Modified)	AT N571 Passive Badge	0.00798 L/min	8 Hours	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
n-Propyl Acetate	NIOSH 1450	150-mg Charcoal Tube	0.01-0.2	18-150	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Pyridine	NIOSH 1613	150-mg Charcoal Tube	0.01-1.0	1 - 10	Undetermined	Should be shipped on ice or equivalent; refrigerate upon receipt.
Selenium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	60 - 2000 / 13 - 2000	Indefinite	Should be stored at room temperature.
Silicon Tetrahydride (Silane)	OSHA CSI	15 mL of 0.01 N KOH in a MGFB	1.0 L/min Max	480 L Max	7 days	Should be stored at room temperature.
Silver	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	60 - 2000 / 250-2000	Indefinite	Should be stored at room temperature.
Sodium	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	30 - 2000 / 30 - 960	Indefinite	Should be stored at room temperature.
Strontium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	25 - 2000 / 10 - 1000	Indefinite	Should be stored at room temperature.
Styrene	NIOSH 1501	150-mg Charcoal Tube	< 1.0	1 - 14	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0289	8 Hrs. Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0137	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00094 (#546) 0.00755 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Sulfuric Acid	NIOSH 7903	600-mg Cleaned Silica Gel	0.2 - 0.5	3 - 100	21 Days	Should be stored at room temperature.
Tetrachloroethylene	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	1.0 - 40	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0283	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0131	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00101 (#546) 0.00583 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Tetrahydrofuran	NIOSH 1609	150-mg Charcoal Tube	0.01 - 0.2	1 - 9	Undetermined	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0372	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0174	4 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00121 (#545) 0.00886 (#546)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Thallium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	50 - 2000 / 25 - 2000	Indefinite	Should be stored at room temperature.
Tolualdehyde	OSHA 1007 (Modified)	AT N571 Passive Badge	0.00524 L/min	8 Hours	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Toluene	NIOSH 1501	150-mg Charcoal Tube	< 0.2	1 - 8	30 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0314	8 Hrs Max.	3 Weeks	Should be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0149	8 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00095 (#546) 0.00735 (#566)	8 Hrs Max.	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Toluene-2,4-Diisocyanate (2,4-TDI)	OSHA 42	37-mm Glass Fiber Filter coated with 1,2PP	1 L/min	15	18 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Toluene-2,6-Diisocyanate	OSHA 42	37-mm Glass Fiber Filter	1 L/min	15	18 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
(2,6-TDI)		coated with 1,2PP				
Toxaphene	NIOSH 5039	37-mm, 0.8-um, MCE Filter	0.2 - 1	2 - 30	14 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
1,1,1-Trichloroethane	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	0.1 - 8	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0309	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Monitor	0.0145	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00108 (#546) 0.0065 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
1,1,2-Trichloroethane	NIOSH 1003	150-mg Charcoal Tube	0.01 - 0.2	2 - 60	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0297	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Monitor	0.0125	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00109 (#546) 0.0065 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Trichloroethylene	NIOSH 1022	150-mg Charcoal Tube	0.01 - 0.2	1 - 30	17 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0311	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0143	4 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00109 (#546) 0.00705 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
Valeraldehyde	OSHA 1007 (Modified)	AT N571 Passive Badge	0.00601 L/min	8 Hrs	28 Days	Should be shipped on ice or equivalent; refrigerate upon receipt.
Vanadium	NIOSH 7300	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 2000	Indefinite	Should be stored at room temperature.
Vinyl Acetate	3M (Modified)	3M Badge (3500 or 3520)	0.0358	8 Hrs Max.	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0163	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N545 or N546 Badge	0.00112 (#546) 0.00811 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Vinyl Chloride	NIOSH 1007	(2) 150-mg Charcoal Tube	0.05	3 - 5	10 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Vinylidene Chloride	NIOSH 1015	150-mg Charcoal Tube	0.01 - 0.2	2.5 - 7	21 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0351	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Badge	0.0123	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00130 (#546) 0.00764 (#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Volatile Organic Compounds	OSHA PV2120	Entech Canister	400ml or 1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
Volatile Organic Compounds	EPA TO-15	Entech Canister	1 L	Grab or Time integrated	30 Days	Should be stored at room temperature.
Xylene isomers	NIOSH 1501	150-mg Charcoal Tube	< 0.2	2 - 23	30 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
	3M (Modified)	3M Badge (3500 or 3520)	0.0273	8 Hrs Max.	3 Weeks	May be shipped on ice or equivalent; refrigerate upon receipt.
	SKC (Modified)	SKC 575-002 Passive Monitor	Isomer specific	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.

Analyte	Method Reference	Sample Media	Sampling Rate	Air Volume	Sample Stability	Preservation
	Assay Technology(Mod)	Assay N546 or N566 Badge	0.00093(#546) 0.00668(#566)	8 Hrs Max.	14 Days	May be shipped on ice or equivalent; refrigerate upon receipt.
Zinc	N7300/OSHA ID-121	37-mm, 0.8-um, MCE Filter	1 - 4	5 - 960 / 5 - 200	Indefinite	Should be stored at room temperature.

## **SECTION 23. HANDLING OF SAMPLES**

Sample management procedures at the laboratory ensure that sample integrity and custody are maintained and documented from sampling/receipt through disposal. This section applies to all samples (environmental, air, industrial hygiene, etc.) received at the laboratory except as noted in individual method SOPs.

### **23.1 Chain of Custody (COC)**

The COC form is the written documented history of any sample and is initiated when bottles are sent to the field, or at the time of sampling. This form is completed by the sampling personnel and accompanies the samples to the laboratory where it is received and stored under the laboratory's custody. The purpose of the COC form is to provide a legal written record of the handling of samples from the time of collection until they are received at the laboratory. It also serves as the primary written request for analyses from the client to the laboratory. The COC form acts as a purchase order for analytical services when no other contractual agreement is in effect. An example of a COC form may be found in Figure 23-1.

#### **23.1.1 Field Documentation**

The information the sampler needs to provide at the time of sampling on the container label is:

- Sample identification
- Date and time
- Preservative

During the sampling process, the COC form is completed and must be legible (see Figure 23-1). This form includes information such as:

- Client name, address, phone number and fax number (if available)
- Project name and/or number
- The sample identification
- Date, time and location of sampling
- Sample collectors name
- The matrix description
- The container description
- The total number of each type of container
- Preservatives used
- Analysis requested
- Requested turnaround time (TAT)
- Any special instructions
- Purchase Order number or billing information (e.g. quote number) if available
- The date and time that each person received or relinquished the sample(s), including their signed name

When the sampling personnel deliver the samples directly to TestAmerica personnel, 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 personnel. 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. When sampling personnel deliver the samples through a common carrier (Fed-Ex, UPS), the CoC relinquished date/time is completed by the field personnel and samples are released to the carrier. Samples are only considered to be received by lab when personnel at the fixed laboratory facility have physical contact with the samples.

**Note:** Independent couriers are not required to sign the COC form. The COC is usually kept in the sealed sample cooler. The receipt from the courier is stored in the project folder.

### **23.1.2 Legal / Evidentiary Chain-of-Custody**

If samples are identified for legal/evidentiary purposes, the Project Manager or Sample Control will enter "Legal ICOC" or "LCOC" into the comments section of Element. Sample control signs the samples into the secure walk-in refrigerator or freezer. Each time the sample is removed or returned to secure storage it is recorded in Element LIMS for that sample. Refer to SOP PE-QAD-026 Internal Chain of Custody Procedures for more detailed information.

## **23.2 Sample Receipt**

Samples are received at the laboratory by designated sample receiving personnel and a unique laboratory project identification number is assigned. Each sample container shall be assigned a unique sample identification number that is cross-referenced to the client identification number such that traceability of test samples is unambiguous and documented. Each sample container is affixed with a durable sample identification label. Sample acceptance, receipt, tracking and storage procedures are summarized in the following sections. SOP No. PE-SMP-001, Sample Receiving describes these procedures.

### **23.2.1 Laboratory Receipt**

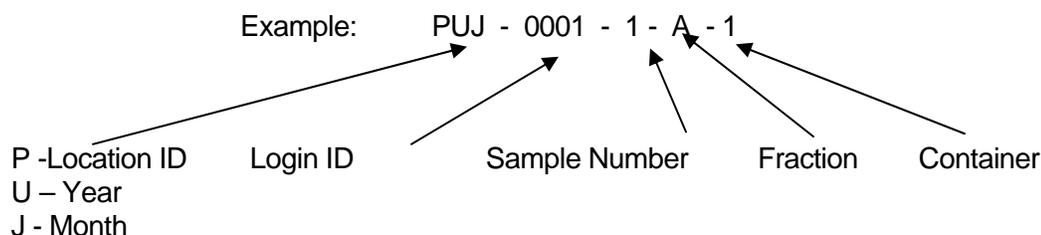
When samples arrive at the laboratory, sample receiving personnel inspect the coolers and samples. The integrity of each sample must be determined by comparing sample labels or tags with the COC and by visual checks of the container for possible damage. Any non-conformance, irregularity, or compromised sample receipt must be documented on a Notification of Discrepancy form and brought to the immediate attention of the client. The COC, shipping documents, documentation of any non-conformance, irregularity, or compromised sample receipt, record of client contact, and resulting instructions become part of the project record.

#### **23.2.1.1 Unique Sample Identification**

All samples that are processed through the laboratory receive a unique sample identification to ensure that there can be no confusion regarding the identity of such samples at anytime. This

system includes identification for all samples, subsamples and subsequent extracts and/or digestates.

The laboratory assigns a unique identification (e.g., Sample ID) code to each sample container received at the laboratory. This Primary ID is made up of the following information (consisting of 7 components):



The above example states that TestAmerica Phoenix Laboratory (Location) received samples in 2011 (Year) in October (Month). Login ID is 0001 (start at 0001 each month). The container code indicates it is the first sample ("1") of the work order and the first fraction ("A") of Sample #1 and the first container of the fraction ("1").

With this system, a client sample can literally be tracked throughout the laboratory in every step from receipt to disposal.

### 23.3 Sample Acceptance Policy

The laboratory has a written sample acceptance policy (Figure 23-2) that clearly outlines the circumstances under which samples shall be accepted or rejected. These include:

- A COC filled out completely;
- Samples must be properly labeled;
- Proper sample containers with adequate volume for the analysis of both environmental and IH samples and necessary QC;
- Samples must be preserved according to the requirements of the requested analytical method or IH Sampling Guide;
- Sample holding times must be adhered to;
- All samples submitted for water 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.

**23.3.1** After inspecting the samples, the sample receiving personnel sign and date the COC form, make any necessary notes of the samples' conditions and store them in appropriate refrigerators or storage locations.

**23.3.2** Sample condition at the time of receipt is documented on the Sample Receipt Forms. A form generated by Element documents general information. A second form, generated by sample receipt personnel documents the verification of sample preservation. This form is not required for samples which do not require chemical preservation (e.g. soil, air) or where verification cannot be performed (e.g. aqueous volatile organics).

**23.3.3** Any deviations from these checks that question the suitability of the sample for analysis, or incomplete documentation as to the tests required will be resolved by consultation with the client. If the sample acceptance policy criteria are not met, the laboratory shall either:

- Retain all correspondence and/or records of communications with the client regarding the disposition of rejected samples, or
- Fully document any decision to proceed with sample analysis that does not meet sample acceptance criteria.

Once sample acceptance is verified, the samples are logged into the LIMS according SOP PE-SMP-001 Sample Control.

#### **23.4 Sample Storage**

In order to avoid deterioration, contamination or damage to a sample during storage and handling, from the time of receipt until all analyses are complete, samples are stored in refrigerators freezers or protected locations suitable for the sample matrix. Aqueous Metal samples and some IH samples are stored unrefrigerated. In addition, samples to be analyzed for volatile organic analytes are stored in separate refrigerators designated for volatile organic parameters only. Samples are never to be stored with reagents, standards or materials that may create contamination.

To ensure the integrity of the samples during storage, refrigerator blanks are maintained in the volatile sample refrigerators and analyzed every two weeks.

Analysts and technicians retrieve the sample container allocated to their analysis from the designated refrigerator and place them on carts, analyze the sample, and return the remaining sample or empty container to the refrigerator from which it originally came. All samples are kept in the refrigerators for two to four weeks after analysis, which meets or exceeds most sample holding times. After two to four weeks the samples are moved to the room temperature sample archive area where they are stored for an additional four weeks before they are disposed of. This eight week holding period allows samples to be checked if a discrepancy or question arises. Special arrangements may be made to store samples for longer periods of time. This extended holding period allows additional metal analyses to be performed on the archived sample and assists clients in dealing with legal matters or regulatory issues.

Access to the laboratory is controlled such that sample storage need not be locked 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.

To minimize exposure to personnel and to avoid potential accidents, hazardous and foreign soil samples are stored in Hazardous Sample bins in an isolated area designated for storage of hazardous samples and foreign soils only. For any sample that is known to be hazardous at the time of receipt or, if after completion of analysis the result exceeds the acceptable regulatory levels, a Hazardous Sample Notification Form must be completed by the analyst.

This form may be completed by Sample Control, Project Managers, or analysts and must be attached to the report. The sample itself is clearly marked with a red tag reading "HAZARDOUS" or "FOREIGN SOIL" and is placed in the Hazardous Samples Bins. A copy of the form must be included with the original COC and Work Order and the original must be given to the Sample Control Custodian. Analysts will present any sample determined to be hazardous after completion of analysis for storage in the assigned area to the Sample Control Custodian.

All hazardous samples are either returned to the client or disposed of appropriately through a hazardous waste disposal firm or via a laboratory waste stream. Depending on the circumstances, clients may be asked to bear the disposal cost if the laboratory is asked to dispose of the hazardous sample. Additional information about handling Hazardous Samples is contained in SOP PE-SMP-001 Sample Control and SOP PE-SFT-001 Sample Disposal and Waste Management. Foreign soil samples are handled according to the procedures in SOP PE-SMP-006 Receiving and Waste Management of Foreign Soils.

### **23.5 Sample Shipping**

In the event that the laboratory needs to ship samples, the samples are placed in a cooler with enough ice to ensure the samples remain just above freezing and at or below 6.0°C during transit. The samples are carefully surrounded by packing material to avoid breakage (yet maintain appropriate temperature). A trip blank is enclosed for those samples requiring water/solid volatile organic analyses (see Note). The chain-of-custody form is signed by the sample control technician and attached to the shipping paperwork. Samples are generally shipped overnight express or hand-delivered by a TestAmerica courier to maintain sample integrity. All personnel involved with shipping and receiving samples must be trained to maintain the proper chain-of-custody documentation and to keep the samples intact and on ice. The Environmental, Health and Safety Manual contains additional shipping requirements.

### **23.6 Sample Disposal**

Samples should be retained for a minimum of 30 days after the project report is sent, however, provisions may be made for earlier disposal of samples once the holding time is exceeded. Some samples are required to be held for longer periods based on regulatory or client requirements (e.g., 60 days after project report is sent). The laboratory must follow the longer sample retention requirements where required by regulation or client agreement. Several possibilities for sample disposal exist: the sample may be consumed completely during analysis, the sample may be returned to the customer or location of sampling for disposal, or the sample may be disposed of in accordance with the laboratory's waste disposal procedures (SOP PE-SFT-001 Sample Disposal and Waste Management). All procedures in the laboratory Environmental, Health and Safety Manual are followed during disposal. Samples are normally maintained in the laboratory no longer than 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's disposal. All documentation and correspondence concerning the disposal decision process must be kept on file.



**Figure 23-2 Sample Acceptance Policy****Sample Acceptance Policy**  
Phoenix Sample Acceptance Policy

All incoming work will be evaluated against the criteria listed below. Where applicable, data from any samples that do not meet the criteria listed below will be noted on the laboratory report defining the nature and substance of the variation. In addition, the client will be notified either by telephone, fax or e-mail ASAP after the receipt of the samples.

- 1) Samples must arrive with labels intact with a Chain of Custody filled out completely. The following information must be recorded.
  - *Client name, address, phone number and fax number (if available)*
  - *Project name and/or number*
  - *The sample identification*
  - *Date, time and location of sampling*
  - *The collectors name*
  - *The matrix description*
  - *The container description*
  - *The total number of each type of container*
  - *Preservatives used*
  - *Analysis requested*
  - *Requested turnaround time (TAT)*
  - *Any special instructions*
  - *Purchase Order number or billing information (e.g., quote number) if available*
  - *The date and time that each person received or relinquished the sample(s), including their signed name.*
  - ***The date and time of receipt must be recorded between the last person to relinquish the samples and the person who receives the samples in the lab. The date and time relinquished/received must be exactly the same.***
    - **Information must be legible**
- 2) Samples must be properly labeled.
  - Use durable labels (labels provided by the Phoenix laboratory are preferred)
  - Include a unique identification number
  - Include sampling date and time & sampler ID
  - Include preservative used
  - Use indelible ink
  - **Information must be legible**
- 3) Proper sample containers with adequate volume for the analysis and necessary QC are required for each analysis requested.
- 4) Samples must be preserved according to the requirements of the requested analytical method. This includes samples (other than water samples for metals analysis) being chilled to below 6° C and above freezing (0°C). **Note:** Samples that are hand delivered to the laboratory immediately after collection may not have had time to cool sufficiently. In this case the samples will be considered acceptable as long as there is evidence that the chilling process has begun (arrival on ice).

- Chemical preservation (pH) will be verified prior to analysis and the project manager will be notified immediately if there is a discrepancy. If analyses will still be performed, all affected results will be flagged to indicate improper preservation.
  - For Volatile Organic analyses in drinking water (Method 524.2). Residual chlorine must be neutralized prior to preservation. If there is prior knowledge that the samples are not chlorinated, state it on the COC and use the VOA vials pre-preserved with HCl. The following are other options for a sampler and laboratory where the presence of chlorine is not known:
    - 1. Test for residual chlorine in the field prior to sampling.
      - If no chlorine is present, the samples are to be preserved using HCl as usual.
      - If chlorine is present, add ascorbic acid prior to adding HCl.
    - 2. Use VOA vials pre-preserved with ascorbic acid and add HCl after filling the VOA vial with the sample.
- 5) Sample Holding Times
- The Phoenix laboratory will make every effort to analyze samples within the regulatory holding time. Samples must be received in the laboratory with enough time to perform the sample analysis. Except for short holding time samples (< 48 hr HT) samples must be received with at least 48 hrs (working days) remaining on the holding time for us to ensure analysis.
  - Analyses that are “field” analyses (e.g., pH, Dissolved Oxygen, Residual Chlorine) will be analyzed within 24 hours from receipt of the samples in the laboratory. Field analysis received after 4:00 PM on Friday or on the weekend will be analyzed no later than the next business day after receipt (Monday, unless a holiday).
- 6) Samples submitted for Volatile Organic analyses must also have a Trip Blank submitted at the same time. TestAmerica’s Phoenix laboratory will supply a blank with the bottle order.
- 7) The project manager will be notified if any sample is received in damaged condition. The Phoenix laboratory will request that a sample be resubmitted for analysis.
- 8) Recommendations for packing samples for shipment.
- Pack samples in Ice rather than “Blue” ice packs.
  - Soil samples should be placed in plastic zip-lock bags. The containers often have dirt around the top and do not seal very well and are prone to intrusion from the water from melted ice.
  - Water samples are best if wrapped with bubble-wrap or paper (newspaper or paper towels work) and then placed in plastic zip-lock bags.
  - Fill extra cooler space with bubble wrap.

Figure 23-3 Sample Receipt Forms



THE LEADER IN ENVIRONMENTAL TESTING

**SAMPLE RECEIPT FORM**

Date/Time: 10/17/2011 10:43:32f

Client Code: 8000

TAL Project Number: PUJ0953

Received By: Peter Floyd

Logged By: Peter Floyd

Sample Temperature: 0.8°C

Samples Received:  On Ice  On Blue Ice  Unchilled

Check All that Apply:				
Analysis	N/A	pH Verified	Additional Preservative Added?	Sample Numbers Needing Adjustment
500ml Amber w/HCL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> HCL	_____
1L Amber w/HCL	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> HCL	_____
Poly w/HNO3	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> HNO3	_____
Poly w/H2SO4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> H2SO4	_____
500ml Amber w/H2SO4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> H2SO4	_____
1L Amber w/H2SO4	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> H2SO4	_____
Poly w/NaOH	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> NaOH	_____
Poly w/ NaOH + Zinc Acetate	<input type="checkbox"/>	<input type="checkbox"/>	<input type="checkbox"/> NaOH + Zinc Acetate	_____

Volatile Soil Samples Received in:  N/A  Brass Sleeves  Glass Jars  Encore  Field Methanol

Other: \_\_\_\_\_

Date	Initials	Sample Number	Comments
10/17/2011		PUJ0953-01	
10/17/2011		PUJ0953-02	
10/17/2011		PUJ0953-03	
10/17/2011		PUJ0953-04	

Reviewed By: cm Date: 10/17/11 Time: \_\_\_\_\_

10/17/2011 10:43:32AM

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TestAmerica  
 Phoenix

Sample Receipt Preservation Verification

Work Order: \_\_\_\_\_  
 Sample Tech: \_\_\_\_\_  
 Date: \_\_\_\_\_

Preservative	Sample Numbers Verified	Sample Numbers with Incorrect Preservation
HNO3 pH ≤ 2		
H2SO4 pH ≤ 2		
NaOH pH ≥ 12		
NaOH+ Zinc Acetate pH ≥ 9		

pH of Incorrect Samples

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

PX-QAD-023A-1/11

## SECTION 24. ASSURING THE QUALITY OF TEST RESULTS

### 24.1 Overview

In order to assure our clients of the validity of their data, the laboratory continuously evaluates the quality of the analytical process. The analytical process is controlled not only by instrument calibration as discussed in Section 20, but also by routine process quality control (QC) measurements (e.g. Blanks, Laboratory Control Samples (LCS) (also known as a Blank Spike (BS)), 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.

Required QC is method/program dependant (EPA methods, TNI, AIHA) and may vary depending on analytes requested. For more definitive information, reference the individual laboratory method SOPs. The following is a generalized discussion of common quality control measures in use in the laboratory.

### 24.2 Controls

Sample preparation or pre-treatment is commonly required before analysis. Typical preparation steps include homogenization, solvent extraction, sonication, acid digestion, distillation, reflux, evaporation, and drying. During these pre-treatment steps, samples are arranged into discreet manageable groups referred to as preparation (prep) batches. Prep batches provide a means to control variability in sample treatment. Control samples are added to each prep batch to monitor method performance and are processed through the entire analytical procedure with investigative/field samples.

### 24.3 Negative Controls

**Table 24-1. Example – Negative Controls**

Control Type	Details
Method Blank (MB)	<p>Are used to assess preparation and analysis for possible contamination during the preparation and processing steps.</p> <p>The specific frequency of use for method blanks during the analytical sequence is defined in the specific standard operating procedure for each analysis. Generally it is 1 for each batch of samples (including IH samples); not to exceed 20 environmental samples.</p> <p>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, sample media (IH Samples), etc.) and is processed along with and under the same conditions as the associated samples.</p> <p>The method blank goes through all of the steps of the process (including as necessary: filtration, clean-ups, etc.).</p>

Control Type	Details
	Reanalyze or qualify associated sample results when the concentration of a targeted analyte in the blank is at or above the reporting limit as established by the method or by regulation, AND is greater than 1/10 of the amount measured in the sample.
Calibration Blanks	Are prepared and analyzed along with calibration standards where applicable. They are prepared using the same reagents that are used to prepare the standards. In some analyses the calibration blank may be included in the calibration curve.
Instrument Blanks	Are blank reagents or reagent water that may be processed during an analytical sequence in order to assess contamination in the analytical system. In general, instrument blanks are used to differentiate between contamination caused by the analytical system and that caused by the sample handling or sample prep process. Instrument blanks may also be inserted throughout the analytical sequence to minimize the effect of carryover from samples with high analyte content.
Trip Blank <sup>1</sup>	Are required to be submitted by the client with each shipment of samples requiring aqueous and solid volatiles analyses (or as specified in the client's project plan).. Additionally, trip blanks may be prepared and analyzed for volatile analysis of air samples, when required by the client. A trip blank may be purchased (certified clean) or is prepared by the laboratory by filling a clean container with pure deionized water that has been purged to remove any volatile compounds. Appropriate preservatives are also added to the container. The trip blank is sent with the bottle order and is intended to reflect the environment that the containers are subjected to throughout shipping and handling and help identify possible sources if contamination is found. The field sampler returns the trip blank in the cooler with the field samples. For IH methods, this may include a media blank submitted with the IH samples.
Field Blanks <sup>1</sup>	Are sometimes used for specific projects by the field samplers. A field blank is 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 and AIHA IH Program)
Equipment Blanks <sup>1</sup>	Are also sometimes created in the field for specific projects. An equipment blank is a sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures. (TNI)
Media Blank <sup>1</sup>	Sorbent or media that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples.
Holding Blanks	Also referred to as refrigerator or freezer blanks, are used to monitor the sample storage units for volatile organic compounds during the storage of VOA samples in the laboratory.

<sup>1</sup> When known, these field QC samples should not be selected for matrix QC as it does not provide information on the behavior of the target compounds in the field samples. Usually, the client sample ID will provide information to identify the field blanks with labels such as "FB", "EB", or "TB."

Evaluation criteria and corrective action for these controls are defined in the specific standard operating procedure for each analysis.

### 24.3.1 Negative Controls for Microbiological Methods

Microbiological Methods utilize a variety of negative controls throughout the process to ensure that false positive results are not obtained. These controls are critical to the validity of the microbiological analyses. Some of these negative controls are:

**Table 24-2. Negative Controls for Microbiology**

<b>Control Type</b>	<b>Details</b>
Sterility Checks (Media)	Are analyzed for each lot of pre-prepared media, ready-to-use media and for each batch of medium prepared by the laboratory.
Filtration Blanks	Blanks are run at the beginning and/or end of each batch depending on the type of water sample. For pre-sterilized single use funnels a sterility check is performed on at least one funnel per lot.
Sterility checks (Sample Containers)	Are performed on at least one container per lot of purchased, pre-sterilized containers. If containers are prepared and sterilized by the laboratory, one container per sterilization batch is checked. Container sterility checks are performed using non-selective growth media.
Sterility Checks (Dilution Water)	Are performed on each batch of dilution water prepared by the laboratory and on each batch of pre-prepared dilution water. All checks are performed using non-selective growth media.
Sterility Checks (Filters)	Are also performed on at least one filter from each new lot of membrane filters using non-selective growth media.

Negative culture controls demonstrate that a media does not support the growth of non-target organisms and ensures that there is not an atypical positive reaction from the target organisms. Prior to the first use of the media, each lot of pre-prepared selective media or batch of laboratory prepared selective media is analyzed with at least one known negative culture control as appropriate to the method.

#### **24.4 Positive Controls**

Control samples (e.g., QC indicators) are analyzed with each batch of samples to evaluate data based upon (1) Method Performance (Laboratory Control Sample (LCS) or Blank Spike (BS)), which entails both the preparation and measurement steps; and (2) Matrix Effects (Matrix Spike (MS) (Matrix spikes are not applicable to air samples) 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/accreditation program (SDW, TNI, AIHA, etc.) and each method within those programs specify the control samples that are prepared and/or analyzed with a specific batch.

Note that frequency of control samples vary with specific regulatory, methodology and project specific criteria. Complete details on method control samples are as listed in each analytical SOP.

##### **24.4.1 Method Performance Control - Laboratory Control Sample (LCS)**

**24.4.1.1** The LCS measures the accuracy of the method in a blank matrix and assesses method performance independent of potential field sample matrix affects in a laboratory batch.

**24.4.1.2** The LCS is prepared from a clean matrix similar to that of the associated samples that is free from target analytes (for example: Reagent water, Ottawa sand, glass beads, etc.) and is processed along with and under the same conditions as the associated samples. The LCS is spiked with verified known amounts of analytes or is 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 LCSs may be processed for solid matrices; final results may be calculated as mg/kg or  $\mu\text{g}/\text{kg}$ , assuming 100% solids and a weight equivalent to the aliquot used for the corresponding field samples, to facilitate comparison with the field samples.

**24.4.1.3** Certified pre-made reference material purchased from a NIST/A2LA accredited vendor may also be used for the LCS when the material represents the sample matrix or the analyte is not easily spiked (e.g. solid matrix LCS for metals, TDS, etc.).

**24.4.1.4** The specific frequency of use for LCS during the analytical sequence is defined in the specific standard operating procedure for each analysis. It is generally 1 for each batch of samples; not to exceed 20 environmental samples. For IH Samples it is generally 1 for each analysis day.

**24.4.1.5** If the mandated or requested test method, or project requirements, do not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample (and Matrix Spike) where applicable (e.g. no spike of pH). However, in cases where the components interfere with accurate assessment (such as simultaneously spiking Chlordane, Toxaphene and PCBs in Method 608), the test method has an extremely long list of components or components are incompatible, at a minimum, a representative number of the listed components (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.

- For methods that have 1-10 target analytes, spike all components.
- For methods that include 11-20 target analytes, spike at least 10 or 80%, whichever is greater.
- For methods with more than 20 target analytes, spike at least 16 components.
- Exception: Due to analyte incompatibility in pesticides, Toxaphene and Chlordane are only spiked at client request based on specific project needs.
- Exception: Due to analyte incompatibility between the various PCB Aroclors, Aroclors 1016 and 1260 are used for spiking for methods 8082 and 608 as they cover the range of all of the Aroclors. Aroclor 1242 is spiked for TO-10. Specific Aroclors may be used by request on a project specific basis.

#### **24.4.2 Positive Controls for Microbiological Methods**

- Each lot of pre-prepared media (including chromofluorogenic reagent) and each batch of laboratory prepared media is tested with a pure culture of known positive reaction.

- In addition, every analytical batch also contains a pure culture of known positive reaction.
- A pure culture of known negative reaction is also tested with each analytical batch to ensure specificity of the procedure.

#### 24.5 Sample Matrix Controls

**Table 24-3. Sample Matrix Control**

Control Type	Details	
Matrix Spikes (MS)	Use	Used to assess the effect sample matrix of the spiked sample has on the precision and accuracy of the results generated by the method used.
	Typical Frequency <sup>1</sup>	At a minimum, with each matrix-specific batch of samples processed, an MS is carried through the complete analytical procedure. Unless specified by the client, samples used for spiking are randomly selected and rotated between different client projects. If the mandated or requested test method does not specify the spiking components, the laboratory shall spike all reportable components to be reported in the Laboratory Control Sample and Matrix Spike. Refer to the method SOP for complete details
	Description	Essentially a sample fortified with a known amount of the test analyte(s).
Surrogate	Use	Measures method performance to sample matrix (organics only).
	Typical Frequency <sup>1</sup>	Are added to all samples, standards, and blanks, for all organic chromatography methods except when the matrix precludes its use or when a surrogate is not available. The recovery of the surrogates is compared to the acceptance limits for the specific method. Poor surrogate recovery may indicate a problem with sample composition and shall be reported, with data qualifiers, to the client whose sample produced poor recovery.
	Description	Are similar to matrix spikes except the analytes are compounds with properties that mimic the analyte of interest and are unlikely to be found in environment samples.
Duplicates <sup>2</sup>	Use	For a measure of analytical precision, with each matrix-specific batch of samples processed, a matrix duplicate (MD or DUP) sample, matrix spike duplicate (MSD), or LCS duplicate (LCSD) is carried through the complete analytical procedure.
	Typical Frequency <sup>1</sup>	Duplicate samples are usually analyzed with methods that do not require matrix spike analysis.
	Description	Performed by analyzing two aliquots of the same field sample independently or an additional LCS.
Internal Standards	Use	Are spiked into all environmental and quality control samples (including the initial calibration standards) to monitor the qualitative aspect of organic and some inorganic analytical measurements.
	Typical Frequency <sup>1</sup>	All organic and ICP methods as required by the analytical method.
	Description	Used to correct for matrix effects and to help troubleshoot variability in analytical response and are assessed after data acquisition. Possible sources of poor internal standard response are sample matrix, poor analytical technique or instrument performance.

<sup>1</sup> See the specific analytical SOP for type and frequency of sample matrix control samples.

<sup>2</sup> LCSDs may not be required except when regulatory agencies or client specifications require them. The recoveries for the spiked duplicate samples must meet the same laboratory established recovery limits as the accuracy QC samples. If an LCSD is analyzed, both the LCS and LCSD must meet the same recovery criteria and be included in the final report. The precision measurement is reported as "Relative Percent Difference" (RPD). Poor precision between duplicates (except LCS/LCSD) may indicate non-homogeneous matrix or sampling.

## **24.6 Acceptance Criteria (Control Limits)**

As mandated by the test method and regulation, each individual analyte in the LCS, MS, or Surrogate Spike is evaluated against the control limits published in the test method. Where there are no established acceptance criteria, the laboratory calculates in-house control limits with the use of control charts or, in some cases, utilizes client project specific control limits. When this occurs, the regulatory or project limits will supersede the laboratory's in-house limits.

**Note:** For methods, analytes and matrices with very limited data (e.g., unusual matrices not analyzed often), interim limits are established using available data or by analogy to similar methods or matrices.

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 are reviewed and updated if necessary approximately every six months). Control limits are established per method (as opposed to per instrument) regardless of the number of instruments utilized.

Laboratory generated Percent (%) 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). (Element LIMS: The system defaults to collecting the previous 3 months data. This time frame should be shortened if there are more than 200 points since the system slows down tremendously. The time frame should be extended if there are not 20-30 points).

- 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.
- In-house limits cannot be any wider than those mandated in a regulated analytical method. Client or contract required control limits are evaluated against the laboratory's statistically derived control limits to determine if the data quality objectives (DQOs) can be achieved. If laboratory control limits are not consistent with DQOs, then alternatives must be considered, such as method improvements or use of an alternate analytical method.
- The lowest acceptable recovery limit will be 10% (the analyte must be detectable and identifiable). Exception: The lowest acceptable recovery limit for Benzidine will be 5% and the analyte must be detectable and identifiable.
- The maximum acceptable recovery limit will be 150%.
- Unless method specified, the maximum acceptable RPD limit will be 35% for waters and 40% for soils. The minimum RPD limit is 20%.

- If either the high or low end of the control limit changes by  $\leq 5\%$  from the previous control limits, the control chart is visually inspected and, using professional judgment, they may be left unchanged if there is no affect on laboratory ability to meet the existing limits.

**24.6.1** 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 SOP PE-QAD-001 Control Limits and Statistical Process Control for additional information.

**24.6.2** A LCS that is within the acceptance criteria establishes that the analytical system is in control and is used to validate the process. Samples that are analyzed with an LCS with recoveries outside of the acceptance limits may be determined as out of control and should be reanalyzed if possible. If reanalysis is not possible, then the results for all affected analytes for samples within the same batch must be qualified when reported. The internal corrective action process (see Section 12) is also initiated if an LCS exceeds the acceptance limits. Sample results may be qualified and reported without reanalysis if:

- The analyte results are below the reporting limit and the LCS is above the upper control limit.
- If the analytical results are above the relevant regulatory limit and the LCS is below the lower control limit.

Or, for TNI work, there is 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

- Marginal exceedances are recovery exceedances between 3 SD and 4 SD from the mean recovery limit (TNI).
- 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.

Though marginal exceedances may be allowed, the data must still be qualified to indicate it is outside of the normal limits.

**24.6.3** If the MS/MSDs do not meet acceptance limits, the MS/MSD and the associated spiked sample is reported with a qualifier for those analytes that do not meet limits. If obvious preparation errors are suspected, or if requested by the client, unacceptable MS/MSDs are

reprocessed and reanalyzed to prove matrix interference. A more detailed discussion of acceptance criteria and corrective action can be found in the lab's method SOPs and in Section 12.

**24.6.4** If a surrogate standard falls outside the acceptance limits, if there is not obvious chromatographic matrix interference, reanalyze the sample to confirm a possible matrix effect. If the recoveries confirm or there was obvious chromatographic interference, results are reported from the original analysis and a qualifier is added. If the reanalysis meets surrogate recovery criteria, the second run is reported (or both are reported if requested by the client). Under certain circumstances, where all of the samples are from the same location and share similar chromatography, the reanalysis may be performed on a single sample rather than all of the samples and if the surrogate meets the recovery criteria in the reanalysis, all of the affected samples would require reanalysis.

## **24.7 Additional Procedures to Assure Quality Control**

**24.7.1** The laboratory has written and approved method SOPs to assure the accuracy of the test method including calibration (see Section 20), use of certified reference materials (see Section 21) and use of PT samples (see Section 15).

**24.7.2** A discussion regarding MDLs, Limit of Detection (LOD) and Limit of Quantitation (LOQ) can be found in Section 19.

**24.7.3** Use of formulae to reduce data is discussed in the method SOPs and in Section 20.

**24.7.4** Selection of appropriate reagents and standards is included in Section 9 and 21.

**24.7.5** A discussion on selectivity of the test is included in Section 5.

**24.7.6** Constant and consistent test conditions are discussed in Section 18.

**24.7.7** The laboratories sample acceptance policy is included in Section 23.

## **SECTION 25. REPORTING RESULTS**

### **25.1 Overview**

The results of each test are reported accurately, clearly, unambiguously, and objectively in accordance with State and Federal regulations as well as client requirements. Analytical results are issued in a format that is intended to satisfy customer and laboratory accreditation requirements as well as provide the end user with the information needed to properly evaluate the results. Where there is conflict between client requests and laboratory ethics or regulatory requirements, the laboratory's ethical and legal requirements are paramount, and the laboratory will work with the client during project set up to develop an acceptable solution. Refer to Section 7.

A variety of report formats are available to meet specific needs.

In cases where a client asks for simplified reports, there must be a written request from the client. There still must be enough information that would show any analyses that were out of conformance (QC out of limits) and there should be a reference to a full report that is made available to the client.

Review of reported data is included in Section 19.

## **25.2      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 on laboratory letterhead, reviewed, and signed by the appropriate Project Manager. At a minimum, the standard laboratory report shall contain the following information:

**25.2.1**      A report title (e.g. Laboratory Report) with a "Sample Result" column header.

**25.2.2**      Each report page printed on company letterhead, which includes the laboratory name, address and telephone number.

**25.2.3**      A unique identification of the report (e.g. report number) and on each page an identification in order to ensure the page is recognized as part of the report and a clear identification of the end.

**Note:**      Page numbers of report are represented as page # of ##. Where the first number is the page number and the second is the total number of pages.

**25.2.4**      A copy of the chain of custody (COC).

- Any CoCs involved with Subcontracting are included.
- In most cases, the applicable COC is not paginated but is an integral part of the report. If the COC is not a paginated portion of the report then there will be a statement on the front of the report to effect of "The Chain of Custody, X page(s), is included and is an integral part of this report.". The number of pages of the CoC (X) is entered into Element so that it is correct for each report
- CoC Exception: For IH laboratory reports, the COC is considered a separate document.
- Any additional addenda to the report must be treated in a similar fashion so it is a recognizable part of the report and cannot accidentally get separated from the report (e.g. Sampling information).

**25.2.5**      The name and address of client and a project name/number, if applicable.

**25.2.6**      Client project manager or other contact.

**25.2.7**      Description and unambiguous identification of the tested sample(s) including the client identification code.

- 25.2.8** Date of receipt of sample, date and time of collection, and date(s) of test preparation and performance, and time of preparation or analysis if the required holding time for either activity is less than or equal to 72 hours.
- 25.2.9** Date reported or date of revision, if applicable.
- 25.2.10** Method of analysis including method code (EPA, Standard Methods, etc); and in the case of IH methods, any modifications to the methods. For Industrial Hygiene reports, test results not covered under AIHA-LAP accreditation must be clearly identified on the final test report.
- 25.2.11** Reporting limits.
- 25.2.12** Method detection limits (if requested).
- 25.2.13** Definition of Data qualifiers and reporting acronyms (e.g. ND).
- 25.2.14** Sample results. Measurement below the reporting limit are reported as < the reporting limit or ND. Results are not reported as "0".
- 25.2.15** For AIHA projects, the final report includes the measured quantitative result of the analysis of any blank samples submitted to the laboratory. Additionally, a statement is included that discloses whether or not the sample results have been corrected for contamination based on the field blank or method blank.
- 25.2.16** QC data consisting of method blank, surrogate, LCS, and MS/MSD recoveries and control limits.
- 25.2.17** Condition of samples at receipt including temperature. This may be accomplished in a narrative or by attaching sample login sheets (Refer to Sec. 25.2.4 – Item 4 regarding additional addenda).
- 25.2.18** A statement expressing the validity of the results, that the source methodology was followed and all results were reviewed for error.
- 25.2.19** A statement to the effect that the results relate only to the items tested and the sample as received by the laboratory.
- 25.2.20** A statement that the report shall not be reproduced except in full, without prior express written approval by the laboratory.
- 25.2.21** A signature and title of the person(s) accepting responsibility for the content of the report and date of issue. Signatories are appointed by the Lab Director.
- 25.2.22** When TNI accreditation is required, the lab shall certify that the test results meet all requirements of TNI or provide reasons and/or justification if they do not.
- 25.2.23** Where applicable, a narrative to the report that explains the issue(s) and corrective action(s) taken in the event that a specific accreditation or certification requirement was not met.

**25.2.24** When soil samples are analyzed, a specific identification as to whether soils are reported on a “wet weight” or “dry weight” basis.

**25.2.25** Appropriate laboratory certification number for the state of origin of the sample, if applicable.

**25.2.26** 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 draft report). A complete report must be sent once all of the work has been completed.

**25.2.27** 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.

**25.2.28** Non-accredited tests shall be clearly identified in the case narrative when claims of accreditation to the TNI standard are made.

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 Reporting Level or Report Type**

The laboratory offers four levels of quality control reporting. Each level, in addition to its own specific requirements, contains all the information provided in the preceding level. The packages provide the following information in addition to the information described above:

- Level I/II is a report with all the features described in Section 25.2 plus summary information; including results for the method blank; percent recovery for laboratory control samples and matrix spike samples; and the RPD values for all MSD and sample duplicate analyses.
- Level III contains all the information supplied in Level I/II; chromatograms, including QC, calibration standards and samples; quantitation reports; initial and continuing calibration information; and copies of bench sheets/instrument printouts where required.
- Level IV is the same as Level III with the addition of multiple sample dilutions; extraction/preparation logs; analysis logs and standard preparation logs.

In addition to the various levels of QC packaging, the laboratory also provides reports in diskette deliverable form. Initial reports may be provided to clients by facsimile. All faxed reports are followed by hardcopy. Procedures used to ensure client confidentiality are outlined in Section 25.6.

#### **25.3.1 Electronic Data Deliverables (EDDs)**

EDDs are routinely offered as part of TestAmerica’s services. TestAmerica Phoenix offers a variety of EDD formats including Excel, ASCII, Dbase, and Access.

EDD specifications are submitted to the IT department by the PM for review and undergo the contract review process. Once the facility has committed to providing data in a specific electronic format, the coding of the format may need to be performed. This coding is

documented and validated. The validation of the code is retained by the IT staff coding the EDD.

EDDs shall be subject to a review to ensure their accuracy and completeness. If EDD generation is automated, review may be reduced to periodic screening if the laboratory can demonstrate that it can routinely generate that EDD without errors. Any revisions to the EDD format must be reviewed until it is demonstrated that it can routinely be generated without errors. If the EDD can be reproduced accurately and if all subsequent EDDs can be produced error-free, each EDD does not necessarily require a review.

#### **25.4 Supplemental Information for Test**

The lab identifies any unacceptable QC analyses or any other unusual circumstances or observations such as environmental conditions and any non-standard conditions that may have affected the quality of a result. This is typically in the form of a footnote or a qualifier and/or a narrative explaining the discrepancy in the front of the report.

Numeric results with values outside of the calibration range, either high or low are qualified as 'estimated'.

Where quality system requirements are not met, a statement of compliance/non-compliance with requirements and/or specifications is required, including identification of test results derived from any sample that did not meet TNI sample acceptance requirements such as improper container, holding time, or temperature.

Where applicable, a statement on the estimated uncertainty of measurements; information on uncertainty is needed when a client's instructions so require.

Opinions and Interpretations - The test report contains objective information, and generally does not contain subjective information such as opinions and interpretations. If such information is required by the client, the Laboratory Director will determine if a response can be prepared. If so, the Laboratory Director will designate the appropriate member of the management team to prepare a response. The response will be fully documented, and reviewed by the Laboratory Director, before release to the client. There may be additional fees charged to the client at this time, as this is a non-routine function of the laboratory.

**Note:** Review of data deliverable packages for submittal to regulatory authorities requires responses to non-conforming data concerning potential impact on data quality. This necessitates a limited scope of interpretation, and this work is performed by the QA Department. This is the only form of "interpretation" of data that is routinely performed by the laboratory.

When opinions or interpretations are included in the report, the laboratory provides an explanation as to the basis upon which the opinions and interpretations have been made. Opinions and interpretations are clearly noted as such and where applicable, a comment should be added suggesting that the client verify the opinion or interpretation with their regulator.

## **25.5 Environmental Testing Obtained From Subcontractors**

If the laboratory is not able to provide the client the requested analysis, the samples would be subcontracted following the procedures outlined in the Corporate SOP on Subcontracting (SOP CA-L-S-002).

Data reported from analyses performed by a subcontractor laboratory are clearly identified as such on the analytical report provided to the client. Results from a subcontract laboratory outside of TestAmerica are reported to the client on the subcontract laboratory's original report stationary and the report includes any accompanying documentation.

## **25.6 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.

**25.6.1** Report deliverable formats are discussed with each new client. If a client requests that reports be faxed or e-mailed, the reports are faxed with a cover sheet or e-mailed with the following note that includes a confidentiality statement similar to the following:

*This material is intended only for the use of the individual(s) or entity to whom it is addressed and may contain information that is privileged and confidential. It is our policy that facsimiles are intended for and should be used for business purposes only. If you are not the intended recipient, or the employee or agent responsible for delivering this material to the intended recipient, you are hereby notified that any dissemination, distribution or copying of this facsimile is strictly prohibited. If you have received this communication in error, please notify the sender. Thank you for your professional consideration and cooperation.*

## **25.7 Format of Reports**

The format of reports is designed to accommodate each type of environmental or industrial hygiene test carried out and to minimize the possibility of misunderstanding or misuse.

## 25.8 Amendments to Test Reports

Corrections, additions, or deletions to reports are only made when justification arises through supplemental documentation. Justification is documented using the laboratory's corrective action system (refer to Section 12).

The revised report is retained on the Archive data server, as is the original report. The revised report is stored in the Archive data server under the sample number followed by "REVISION". The revised report will have the word "revised" or "amended" next to the date rather than the word "Issued" or "Reported".

When the report is re-issued, a notation of "report reissued" is placed on the case narrative/signature page of the report in the Comments or Additional Information section of the Case Narrative with a brief explanation of reason for the re-issue and a reference back to the last final report generated. *For Example: Report was revised on 11/3/08 to include toluene in sample NQA1504 per client's request. This final report replaces the final report generated on 10/27/08 at 10:47am.*

Note: Re-issued or revised Industrial Hygiene reports are generated with the current date the report is issued. Information concerning the revision is clearly stated in the Case Narrative and references the original report date.

## 25.9 Policies on Client Requests for Amendments

### 25.9.1 Policy on Data Omissions or Reporting Limit Increases

Fundamentally, our policy is simply to not omit previously reported results (including data qualifiers) or to not raise reporting limits and report sample results as ND. This policy has few exceptions. Exceptions are:

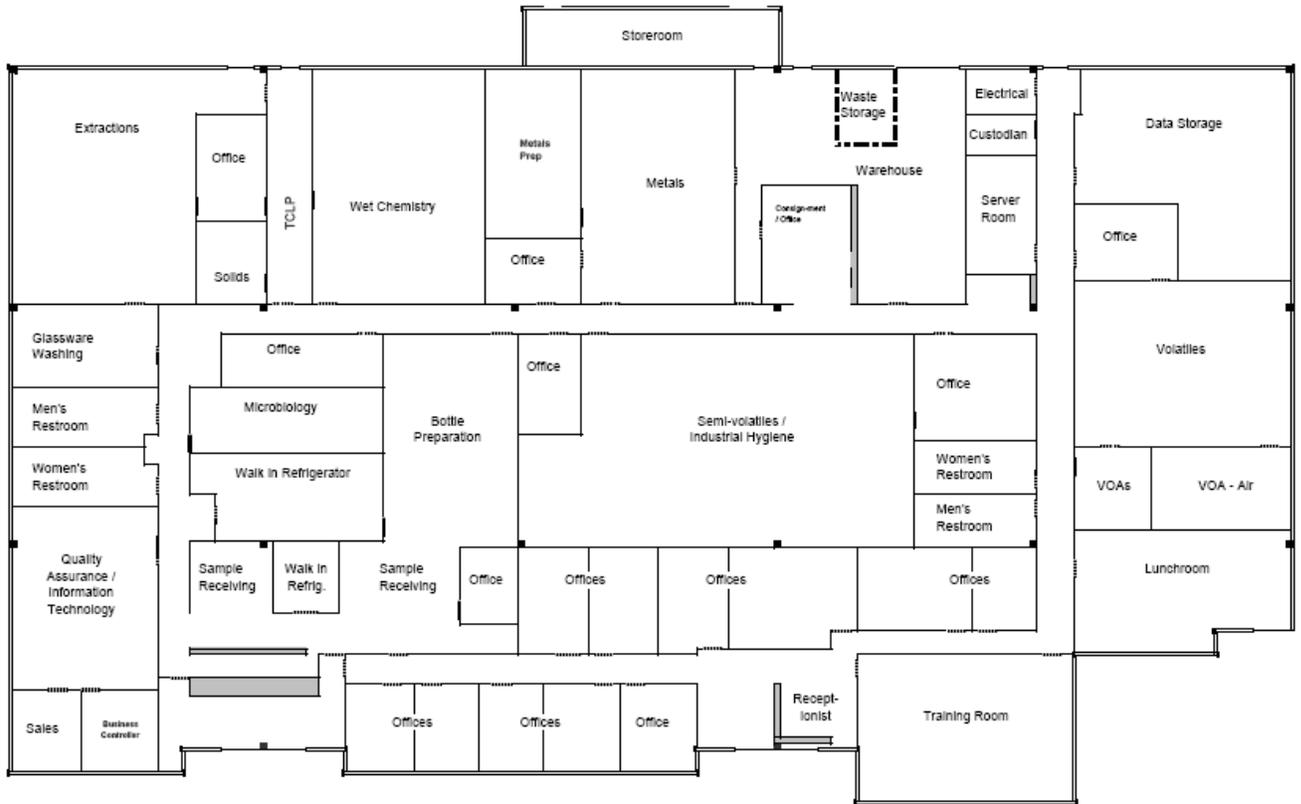
- Laboratory error.
- Sample identification is indeterminate (confusion between COC and sample labels).
- An incorrect analysis (not analyte) was requested (e.g., COC lists 8315 but client wanted 8310). A written request for the change is required.
- Incorrect limits reported based on regulatory requirements.
- The requested change has absolutely 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.

### 25.9.2 Multiple Reports

TestAmerica does not issue multiple reports for the same workorder where there is different information on each report (this does not refer to copies of the same report) unless required to meet regulatory needs and approved by QA.

## Appendix 1. Laboratory Floor Plan

TestAmerica Phoenix Laboratory Floor Plan



## Appendix 2. Glossary/Acronyms

### Glossary:

Acceptance Criteria: Specified limits placed on characteristics of an item, process, or service defined in requirement documents. (ASQC)

Accreditation: The process by which an agency or organization evaluates and recognizes a laboratory as meeting certain predetermined qualifications or standards, thereby accrediting the laboratory.

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)

Analytical Uncertainty: A subset of Measurement Uncertainty that includes all laboratory activities performed as part of the analysis. (TNI)

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 laboratory accreditation). (TNI)

Audit: A systematic and independent examination of facilities, equipment, personnel, training, procedures, record-keeping, data validation, data management, and reporting aspects of a system to determine whether QA/QC and technical activities are being conducted as planned and whether these activities will effectively achieve quality objectives. (TNI)

Batch: Environmental samples which are prepared and/or analyzed together with the same process and Environmental samples that 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 (1) to twenty (20) environmental samples of the same quality systems 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 twenty-four (24) hours. An **analytical batch** is composed of prepared environmental samples (extracts, digestates or concentrates) which are analyzed together as a group. An analytical batch can include prepared samples originating from various quality system matrices and can exceed twenty (20) samples. (TNI)

Bias: The systematic or persistent distortion of a measurement process, which causes errors in one direction (i.e., the expected sample measurement is different from the sample’s true value). (TNI)

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)

Calibration: A set of operations that establish, under specified conditions, the relationship between values of quantities indicated by a measuring instrument or measuring system, or values represented by a material measure or a reference material, and the corresponding values realized by standards. (TNI)

1) In calibration of support equipment the values realized by standards are established through the use of reference standards that are traceable to the International System of Units (SI).

2) In calibration according to methods, the values realized by standards are typically established through the use of Reference Materials that are either purchased by the laboratory with a certificate of analysis or purity, or prepared by the laboratory using support equipment that has been calibrated or verified to meet specifications.

Calibration Blank (CB) A volume of reagent water. The results must fall below the reporting level, the MDL, or a multiplier of the MDL.

Calibration Curve: The graphical relationship between the known values, such as concentrations, of a series of calibration standards and their instrument response. (NELAC)

Calibration Standard (CAL): A substance or reference material used to calibrate an instrument (QAMS), usually prepared from the primary dilution standard solution(s) or stock standard solutions.

Certified Reference Material (CRM): A reference material accompanied by a certificate, having a value, measurement uncertainty, and stated metrological traceability chain to a national metrology institute. (TNI)

Chain of Custody: **Chain of Custody (COC) Form**: Record that documents the possession of the samples from the time of collection to receipt in the laboratory. This record generally includes: the number and types of containers; the mode of collection; the collector; time of collection; preservation; and requested analyses. (TNI)

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.

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. TNI and its representatives agree to safe guarding 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: (TNI)

- Second column confirmation
- Alternate wavelength
- Derivatization
- Mass spectral interpretation
- Alternative detectors or
- Additional Cleanup procedures

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)

Continuing Calibration Verification Standard (CCV): A CAL solution which is analyzed after every tenth field sample analysis, not including QC samples, which verifies the previously established calibration curve and confirms accurate analyte quantitation for the previous field samples analyzed.

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. The analyst will most frequently be the one to identify the need for this action as a result of calibration checks and QC sample analysis. No significant action is taken to change behavior, process or procedure.

Corrective Action: The action taken to eliminate the causes of an existing nonconformity, defect or other undesirable situation in order to prevent recurrence. (ISO 8402)

Data Audit: A qualitative and quantitative evaluation of the documentation and procedures associated with environmental measurements to verify that the resulting data re of acceptable quality (i.e., that they meet specified acceptance criteria).

Data Reduction: The process of transforming the number of data items by arithmetic or statistical calculations, standard curves, and concentration factors, and collation into a more useable form. (TNI)

Deficiency: An unauthorized deviation from acceptable procedures or practices, or a defect in an item. (ASQC)

Demonstration of Capability: A procedure to establish the ability of the analyst to generate analytical results of acceptable accuracy and precision. (TNI)

Detection Limit: See LOD

Document Control: The act of ensuring that documents (and revisions thereto) are proposed, reviewed for accuracy, approved for release by authorized personnel, distributed properly, and controlled to ensure use of the correct version at the location where the prescribed activity if performed. (ASQC)

Duplicate Analyses: The analyses or measurements of the variable of interest performed identically on two subsamples of the same sample. The results from duplicate analyses are used to evaluate analytical or measurement precision but not the precision of sampling, preservation or storage internal to the laboratory. (EPA-QAD)

Equipment Blank: Sample of analyte-free media which has been used to rinse common sampling equipment to check effectiveness of decontamination procedures.

External Standard Calibration: Calibrations for methods that do not utilize internal standards to compensate for changes in instrument conditions.

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 Accreditation: Those matrix, technology/method, and analyte combinations for which the accreditation body offers accreditation.

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)

Initial Calibration Standards (ICAL): A series of CAL solutions used to initially establish instrument calibration and develop calibration curves.

Initial Calibration Verification Standard (ICV): A CAL solution, which is analyzed initially prior to any field sample analysis, which verifies the previously established calibration curve.

Initial Demonstration of Capability (IDC): A procedure to establish the ability of the analyst to generate acceptable accuracy and precision.

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 as a reference for evaluating and controlling the precision and bias of the applied analytical test method. (TNI)

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 Detection Limit (IDL): The minimum amount of a substance that can be measured with a specified degree of confidence that the amount is greater than zero using a specific instrument. The IDL is associated with the instrumental portion of a specific method only, and sample preparation steps are not considered in its derivation. The IDL is a statistical estimation at a specified confidence interval of the concentration at which the relative uncertainty is  $\pm 100\%$ . The IDL represents a range where qualitative detection occurs on a specific instrument. Quantitative results are not produced in this range.

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 (LCS): **(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 of the procedure unless otherwise noted in a reference method. 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.

Laboratory Control Sample Duplicate (LSCD): A second 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.

An LSCD 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.

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(s) of Detection (LOD) [a.k.a., Method Detection Limit (MDL)]: A laboratory's estimate of the minimum amount of an analyte in a given matrix that an analytical process can reliably detect in their facility. (TNI)

Manager (however named): The individual designed as being responsible for the overall operation, all personnel, and the physical plant of the environmental laboratory. A Department Manager may report to the manager. In some cases, the Department Manager 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): A sample prepared, taken through all sample preparation and analytical steps of the procedure unless otherwise noted in a referenced method, by adding a known amount of target analyte to a specified amount of sample for which an independent test result 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 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.

Media Blank: Sorbent or media, that is treated exactly as a sample including exposure to all glassware, equipment, solvents, filtration and reagents that are used with other samples.

Method Blank (also known as Laboratory Reagent 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.

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)

Method Detection Limit Verification: The validity of the MDL is verified by analysis of the analyte(s) in a QC sample in each matrix. This QC sample shall contain the analyte at no more than 3X the LOD for single analyte tests and 4X the LOD for multiple analyte tests. This verification is performed on every instrument that is to be used for analysis of samples and reporting of data. The validity of the LOD is verified as part of the LOD determination process

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)

Non-conformance: An indication, judgment, or state of not having met the requirements of the relevant specifications, contract, or regulation.

Performance Audit:

The routine comparison of independently obtained qualitative and quantitative measurement system data with routinely obtained data in order to evaluate the proficiency of an analyst or laboratory. (NELAC)

Positive Control: Measures taken to ensure that a test and/or its components are working properly and producing correct or expected results from positive test subjects. (TNI)

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: Any conditions under which a sample must be kept in order to maintain chemical and/or biological integrity prior to analysis. (TNI)

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. (TNI)

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. (TNI)

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. (TNI)

Quality Assurance: An integrated system of management activities involving planning, implementation, assessment, reporting and quality improvement to ensure that a process, item, or service is of the type of quality needed and expected by the client. (TNI)

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 that measures the attributes and performance of a process, item, or service against defined standards to verify that they meet the stated requirements established by the customer; operational techniques and activities that are used to fulfill requirements for quality; also the system of activities and checks used to ensure that measurement systems are maintained within prescribed limits, providing protection against “out of control” conditions and ensuring that the results are of acceptable quality. (TNI)

Quality Control Sample: A sample used to assess the performance of all or a portion of the measurement system. One of any number of samples, such as Certified Reference Materials, a quality system matrix fortified by spiking, or actual samples fortified by spiking, intended to demonstrate that a measurement system or activity is in control. (TNI)

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 activities. (TNI)

Quantitation Limits: See LOQ

Raw Data: The documentation generated during sampling and analysis. This documentation includes, but is not limited to, field notes, electronic data, magnetic tapes, untabulated sample results, QC sample results, print outs of chromatograms, instrument outputs, and handwritten records. (TNI)

Record Retention: The systematic collection, indexing and storing of documented information under secure conditions.

Reference Material: Material or substance one or more properties of which are sufficiently well established to be used for the calibration of an apparatus, the assessment of a measurement method, or for assigning values to materials. (ISO Guide 30-2.1)

Reference Standard: Standard used for the calibration of working measurement standards in a given organization or a given location. (TNI)

Relative Percent Difference (RPD): The difference between two values divided by the average of the values as expressed as a percent, used to determine the closeness of two related values.

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)

Sampling: Activity related to obtaining a representative sample of the object of conformity assessment, according to a procedure.

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: The ability to analyze, distinguish, and determine a specific analyte or parameter from another component that may be a potential interferent or that may behave similarly to the target analyte or parameter within the measurement system. (TNI)

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.

Standard: The document describing the elements of laboratory accreditation that has been developed and established within the consensus principles of standard setting and meets the approval requirements of standard adoption organizations procedures and policies. (TNI)

Standard Operating Procedures (SOPs): A written document which details the method for an operation, analysis, or action, with thoroughly prescribed techniques and steps. SOPs are officially approved as the methods for performing certain routine or repetitive tasks. (TNI)

Storage Blank: A blank matrix stored with field samples of a similar matrix (volatiles only) that measures storage contribution to any source of contamination.

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 Manager: A member of the staff of an environmental laboratory who exercises actual day-to-day supervision of laboratory operations for the appropriate fields of accreditation and reporting of results. The Technical Manager must meet TNI requirements for education and experience. The Department Manager acts as a Technical Director designee.

Technology: A specific arrangement of analytical instruments, detection systems, and/or preparation techniques.

Test: A technical operation that consists of the determination of one or more characteristics or performance of a given product, material, equipment, organism, physical phenomenon, process, or service according to a specified procedure. The result of a test is normally recorded in a document sometimes called a test report or a test certificate. (ISO/IEC Guide 2-12.1, amended)

Test Method: An adoption of a scientific technique for a specific measurement problem, as documented in a laboratory SOP. (NELAC)

Traceability: The ability to trace the history, application, or location of an entity by means of recorded identifications. In a calibration sense, traceability relates measuring equipment to national or international standards, primary standards, basic physical constants or properties, or reference materials. In a data collection sense, it relates calculations and data generated throughout the project back to the requirements for the quality of the project. (TNI)

Trip Blank: A blank matrix placed in a sealed container at the laboratory that is shipped, held unopened in the field, and returned to the laboratory in the shipping container with the field samples.

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.

Validation: The process of substantiating specified performance criteria. (EPA-QAD)

**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  
ICP/MS – ICP/Mass Spectrometry  
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  
MDLV - Method Detection Limit Verification  
MS – Matrix Spike  
MSD – Matrix Spike Duplicate  
MSDS - Material Safety Data Sheet  
NELAP - National Environmental Laboratory Accreditation Program  
PT – Performance Testing  
TNI – The NELAC Institute  
QAM – Quality Assurance Manual  
QA/QC – Quality Assurance / Quality Control  
QAPP – Quality Assurance Project Plan  
QCS - Quality Control Sample  
RF – Response Factor  
RPD – Relative Percent Difference  
RSD – Relative Standard Deviation  
SD – Standard Deviation  
SOP: Standard Operating Procedure  
TAT – Turn-Around-Time  
VOA – Volatiles  
VOC – Volatile Organic Compound

**Appendix 3.****Laboratory Certifications, Accreditations, Validations**

TestAmerica Phoenix 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
American Industrial Hygiene Association	154268
Arizona Department of Health Services	AZ0728
California ELAP	2704
California NELAP	01109CA
Nevada	AZ010302009
New York	11898
Oregon (ORELAP)	AZ100001
US Department of Agriculture	P330-10-00310

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.

**Appendix 4.  
Methods Performed**

**Preparation Only Methods**

Method	Aqueous	Solid	Waste	Air
<b>Organics</b>				
EPA 1311	X	X	X	
EPA 3500B	X		X	
EPA 3510C	X		X	
EPA 3520C	X		X	
EPA 3600C	X	X	X	
EPA 3665A H2SO4/Permanganate	X	X	X	
EPA 3545 PFE		X		
EPA 3580A	X	X	X	
EPA 5000	X	X	X	
EPA 5030 B & C	X	X	X	
EPA 5035 & 5035A		X	X	
<b>Inorganics</b>				
EPA 1311	X	X	X	
EPA 1312	X	X	X	
EPA 3005A	X			
EPA 3010A	X		X	
EPA 3020A	X		X	
EPA 3050B		X	X	

**Organics Methods Performed**

<b>Parameter</b>	<b>Method</b>	<b>Aqueous</b>	<b>Solid</b>	<b>Waste</b>	<b>Air</b>
<b>Volatile Organics (VOC)</b>	EPA 8260B	X	X	X	X
	EPA 524.2	X			
	EPA 624	X		X	
	EPA TO-15				X
	EPA TO-17				X
<b>VOC Aromatic &amp; MTBE</b>	EPA 8021B	X	X	X	X
<b>Base Neutrals and Acids (BNAs)</b>	EPA 8270C	X	X	X	
	EPA 625	X			
<b>Organochlorine Pesticides</b>	EPA 8081A	X	X	X	
	EPA 608	X		X	
	EPA 625				
	EPA TO-10A				X
<b>Chlorinated Herbicides</b>	EPA 8151A	X	X	X	
<b>Organophosphorus Pesticides</b>	EPA 8141A	X	X	X	
	EPA 1657	X		X	
	EPA TO-10A				X
<b>PCBs</b>	EPA 8082	X	X	X	
	EPA TO-10A				X
<b>Petroleum Hydrocarbons</b>	ADHS 8015AZ R1		X		
<b>Diesel Range Organics</b>	EPA 8015B/8015D	X	X	X	
<b>Gasoline Range Organics</b>	EPA 8015B/8015D	X	X	X	X
<b>PAHs</b>	EPA 8310	X	X	X	
<b>Formaldehyde</b>	EPA TO-11A				X

**Metals Methods Performed**

Parameter	Methods	Aqueous	Solid	Waste	Air
Trace Metals	EPA 200.7	X			
	EPA 200.8	X			
	EPA 6010B	X	X	X	
	EPA 6020	X	X	X	
	NIOSH 7300				X
Hardness	SM 2340B	X			
	EPA 200.7	X			
Mercury	EPA 245.1	X			
	EPA 7470A	X		X	
	EPA 7471A		X	X	
	NIOSH 6009				X

**Microbiology Methods Performed**

Parameter	Method	Aqueous	Solid	Waste	Air
Fecal Coliform by Mtf	SM 9221E	X	X	X	
Fecal Coliform by Membrane Filtration	SM 9222D	X			
Heterotrophic Bacteria	SIMPLATE	X			
Total Coliforms & E. Coli by Colilert	SM 9223B	X			
Total Coliform by Mf	SM 9221B & C	X			
E. Coli (not for NPDES)	SM9221F	X			

## Inorganics Methods Performed

Parameter	Method	Aqueous	Solid	Waste	Air
n-Hexane Extractables	9070A	X			
SGT treated n-Hexane Extractables	9070A	X			
Alkalinity (Carbonate, Bicarbonate, Total)	SM 2320B	X			
Ammonia	SM 4500 NH3 D	X			
Bromide	EPA 300.0	X			
	EPA 9056	X	X	X	
Carbon, Total Organic	SM 5310B	X			
	EPA 9060A	X		X	
Carbon, Dissolved Organic	SM 5310B	X			
Chloride	EPA 300.0	X			
	EPA 9056	X	X	X	
Chlorine, Total Residual	HACH 8167	X			
Chromium, Hexavalent	SM 3500 CR D	X	X		
Corrosivity	SM 2330B	X			
Conductivity	SM 2510B	X			
	EPA 9050A	X		X	
Cyanide, Total	SM 4500 CN E	X	X	X	
	SM 4500CN B C	X	X	X	
	EPA 9010C		X	X	
	EPA 9013		X	X	
	EPA 9014	X	X	X	
Cyanide, Amenable	SM 4500 CN G	X			
Demand, Biological Oxygen	SM 5210B	X		X	
Demand, Carbonaceous (CBOD)	SM 5210B	X		X	
Demand, Chemical Oxygen	SM 5220D	X		X	
Dissolved Oxygen	SM 4500-O G				
Fluoride	EPA 300.0	X	X		
	EPA 9056	X	X	X	
	SM 4500 F C	X			
Flashpoint	EPA 1010A	X		X	
	EPA 1030		X		
n-Hexane Extractable Materials	EPA 1664A	X			
	EPA 9060A	X		X	
Silica Gel Treated n-	EPA 1664A	X			

**Inorganics Methods Performed**

<b>Parameter</b>	<b>Method</b>	<b>Aqueous</b>	<b>Solid</b>	<b>Waste</b>	<b>Air</b>
<b>Hexane Extractable Materials</b>	EPA 9060A	X		X	

**Inorganics Methods Performed**

<b>Nitrate</b>	EPA 300.0	X			
	EPA 9056	X	X	X	
<b>Nitrate &amp; Nitrite</b>	EPA 300.0	X			
	EPA 9056	X	X	X	
<b>Nitrite</b>	EPA 300.0	X			
	EPA 9056	X	X		
	SM 4500 NO2 B	X			
<b>Total Kjeldahl Nitrogen Total Kjeldahl Nitrogen (NV Only)</b>	SM 4500 NH3 D	X			
	SM 4500 Norg C	X			
<b>Orthophosphate</b>	EPA 300.0	X			
	EPA 9056	X	X	X	
<b>Paint Filter Liquids Test</b>	EPA 9095B	X		X	X
<b>Perchlorate</b>	EPA 314.0	X			
<b>pH</b>	SM 4500 H+ B	X			
	EPA 9040B	X			
	EPA 9041A	X			
	EPA 9045D		X	X	
<b>Phosphorus, Total</b>	SM 4500 P B E	X	X	X	
<b>Solids, Total</b>	SM 2540B	X		X	
<b>Solids, Total Dissolved</b>	SM 2540C	X		X	
<b>Solids, Total Suspended</b>	SM 2540D	X		X	
<b>Solids, Total Volatile</b>	EPA 160.4	X		X	
<b>Settleable Solids</b>	SM 2540F	X			
<b>Solids, Total, Fixed and Volatile</b>	SM 2540G	X	X		
<b>Sulfate</b>	EPA 300.0	X			
	EPA 9056	X	X	X	
<b>Sulfide</b>	SM 4500 S D	X			
<b>Temperature</b>	SM 2550	X			
<b>Turbidity</b>	EPA 180.1	X			

Industrial Hygiene Testing Performed		
Instrumentation	Technology/Detector	Method
Gas Chromatography	GC / FID	NIOSH 1003 (mod)
		NIOSH 1005 (mod)
		NIOSH 1010 (mod)
		NIOSH 1015 (mod)
Gas Chromatography	GC / FID	NIOSH 1022 (mod)
		NIOSH 1300 (mod)
		NIOSH 1400 (mod)
		NIOSH 1401 (mod)
Gas Chromatography	GC / FID	NIOSH 1405 (mod)
		NIOSH 1450 (mod)
		NIOSH 1457 (mod)
		NIOSH 1500 (mod)
Gas Chromatography	GC / FID	NIOSH 1501 (mod)
		NIOSH 1602 (mod)
		NIOSH 1604 (mod)
		NIOSH 1609 (mod)
Gas Chromatography	GC / FID	NIOSH 1605 (mod)
		NIOSH 1615 (mod)
		NIOSH 1610 (mod)
		In-house method for 4-PCH
Gas Chromatography	GC / FID	OSHA 7 (mod)
		NIOSH 2000 (mod)
		NIOSH 1403 (mod)
		NIOSH 2551 (mod)
Gas Chromatography	GC / FID	NIOSH 1550 (mod)
		OSHA 48 (mod)
		NIOSH 2546 (mod)
		NIOSH 1611 (mod)
Gas Chromatography	GC / FID	NIOSH 1613 (mod)
		NIOSH 1606 (mod)
		NIOSH 1007 (mod)
		NIOSH 5523
Gas Chromatography	GC/ECD	NIOSH 5039 (mod)
		NIOSH 5503
		NIOSH 5600
		OSHA PV 2120 (mod)
Gas Chromatography (Diffusive Samplers)	GC / FID	3M 3500
		3M 3520
		OSHA 111 (mod)
		OSHA 1005 (mod)
Gas Chromatography	GC / FID	OSHA 1004 (mod)
		OSHA 7 (mod)
		SKC

<b>Industrial Hygiene Testing Performed</b>		
<b>Instrumentation</b>	<b>Technology/Detector</b>	<b>Method</b>
		Assay Technologies OSHA 69 (mod) OSHA 1001 (mod) OSHA 1002 (mod)
Ion Chromatography (IC)		NIOSH 6013 OSHA ID-215 NIOSH 7903 (mod)
Liquid Chromatography	HPLC/ UV	NIOSH 2016 (mod) OSHA 1007 OSHA42 OSHA 47 OSHA 64 (mod) NIOSH 2532 (mod) Using Assay Technologies 571 Passive monitor Formaldehyde, Carbonyls by HPLC EPA TO-11A EPA IP6A & EPA IP6C NIOSH 5506 (mod)
	HPLC/ FL	NIOSH 5506 (mod)
Atomic Absorption	CVAA	NIOSH 6009 OSHA ID-140
Inductively-Couple Plasma	ICP/MS	NIOSH 7300 (mod) OSHA ID-121 (mod) OSHA ID-125G (mod) NIOSH 7303 (mod)
	ICP/AES	NIOSH 7300 (mod) OSHA ID-121 (mod) OSHA ID-125G (mod) NIOSH 7303 (mod) NIOSH 9100 (mod) NIOSH 9102 (mod) OSHA 121 (mod) OSHA 125G (mod) OSHA CSI Method for Silane OSHA 1003 (mod) NIOSH 6006 (mod)
Spectrometry	UV/Vis (Colorimetric)	NIOSH 7600 (mod) H2S Radiello NIOSH 6010 (mod)
Miscellaneous	Gravimetric	NIOSH 0500 (mod) NIOSH 0600 (mod) NIOSH 5000 (mod)
	Ion Selective Electrode	OSHA ID-188